



## Supporting Information

for

### Application of chiral 2-isoxazoline for the synthesis of *syn*-1,3-diol analogs

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### Experimental procedures and characterization data

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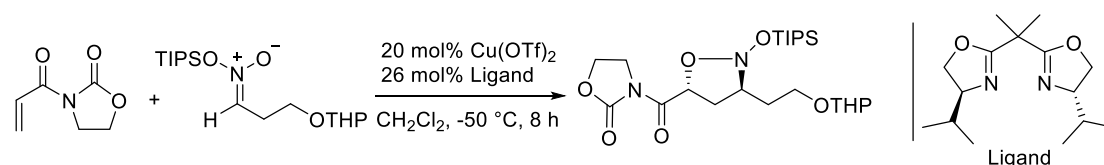
## General information

All glassware for reactions using anhydrous solvents were dried under high vacuum (<0.1 torr) using a heat gun. General Schlenk techniques were applied for addition and transfer operations. Commercial reagents and solvents were used as received unless otherwise noted. THF was distilled over sodium benzophenone ketyl under N<sub>2</sub>. CH<sub>2</sub>Cl<sub>2</sub> was distilled over CaH<sub>2</sub> under N<sub>2</sub>. Thin-layer chromatography was performed on precoated silica gel (0.2–0.25 mm thickness) plates with fluorescent indicator 254 nm. The plate was visualized with 254 nm UV lamp, PMA or KMnO<sub>4</sub> stain. Column chromatography was performed on 200–300 mesh silica gel.

<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded at 400 (or 600) MHz and 100 (or 150) MHz, respectively. Chemical shifts of <sup>1</sup>H NMR and <sup>13</sup>C NMR were referenced to TMS (δ = 0) and chloroform (δ = 77.16), respectively. Chemical shifts of <sup>11</sup>B NMR (128 MHz) were referred to BF<sub>3</sub>·Et<sub>2</sub>O (δ = 0). The following abbreviations were used to denote the multiplicity of each peak: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), m (multiplet). HPLC was performed at room temperature. Specific rotation was measured using the 589 nm D-line of sodium lamp and a quartz cell with 10 cm path length. The X-ray diffraction experiment was conducted using CuKα radiation.

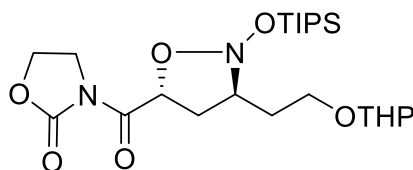
## 1. Preparation of compounds 1–21

### *N*-{(3*S*,5*R*)-[3-(2-(2-Tetrahydropyranyloxy)ethyl)-2-triisopropylsilyloxy-5-isoxazolidinyl]carbonyl}-1,3-oxazolidin-2-one (1)



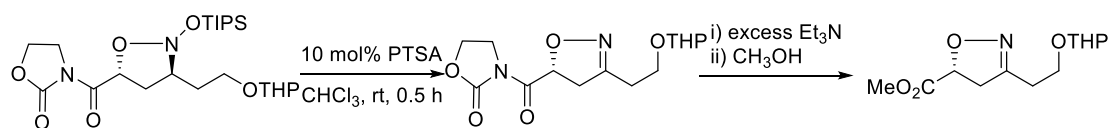
To a dry Schlenk tube were added Cu(OTf)<sub>2</sub> (144 mg, 0.4 mmol), chiral bisoxazoline (139 mg, 0.52 mmol) and anhydrous CH<sub>2</sub>Cl<sub>2</sub> (4 mL) under N<sub>2</sub>. After stirring at room temperature for 2 h, a clear solution was formed, which was cooled to -50 °C and *N*-acryloyl-1,3-oxazolidin-2-one (282 mg, 2 mmol) was added. After stirring for 30 min, a solution of the silyl nitronate (3.0 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (6 mL) was added. The mixture was stirred for 8 h at -50 °C and monitored by TLC. After

the reaction was completed, the product was purified by silica gel chromatography.

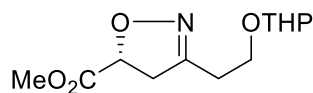


**1:** Yellow oil (923 mg, 95% yield),  $R_f = 0.40$  (1:1 hexanes/AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 5.77–5.74 (m, 1H,  $\text{CH}_2\text{CHO}$ ), 4.53 (s, 1H,  $\text{OCHO}$ ), 4.44 (t,  $J = 8.0$  Hz, 2H,  $\text{CH}_2\text{O}$ ), 4.03–3.99 (m, 2H,  $\text{CH}_2\text{O}$ ), 3.79–3.74 (m, 2H,  $\text{OCH}_2\text{CH}_2$ ), 3.47–3.37 (m, 3H,  $\text{NCH}$  and  $\text{NCH}_2$ ), 2.75–2.66 (m, 1H,  $\text{CHCH}_2\text{CH}$ ), 2.31–2.27 (m, 1H,  $\text{CHCH}_2\text{CH}$ ), 2.17–2.12 (m, 1H,  $\text{CH}_2\text{CH}_2$ ), 1.84–1.79 (m, 2H,  $\text{CH}_2\text{CH}_2$  and  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 1.68–1.49 (m, 6H,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 1.24–1.15 (m, 3H,  $\text{SiCH}$ ), 1.07–1.01 (m, 18H,  $\text{SiCH}(\text{CH}_3)_2$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 170.8, 153.1, 98.9, 98.9, 77.4, 77.2, 69.9, 69.8, 65.2, 62.8, 62.5, 62.3, 42.6, 35.6, 35.5, 30.7, 30.6, 29.9, 29.8, 25.5, 19.6, 19.5, 18.1, 18.0, 12.2; IR ( $\text{cm}^{-1}$ ): 3544, 2942, 2867, 2725, 2249, 1780, 1704, 1464, 1386, 1275, 1133, 1035, 883, 806, 677; MS (ESI): calculated for  $\text{C}_{23}\text{H}_{42}\text{N}_2\text{O}_7\text{Si}$   $[\text{M}+\text{Na}]^+$  509.2659, found 509.2659.

**{(5R)-[3-(2-(2-Tetrahydropyranyloxy)ethyl)]-2-isoxazolin-5-yl}carboxylic acid methyl ester (3)**



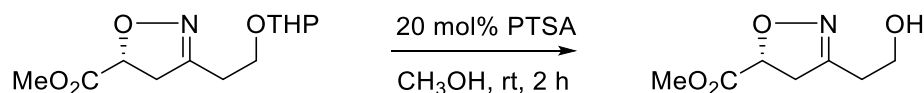
To a solution of **1** (0.86 g, 1.78 mmol) in  $\text{CHCl}_3$  (15 mL) was added PTSA (31 mg, 0.178 mmol) at  $0^\circ\text{C}$ . The mixture was allowed to warm to room temperature and stirred until complete consumption of the starting material (0.5 h). Vacuum was applied to remove the solvent before  $\text{Et}_3\text{N}$  (5 mL) was added. After stirring for 5 min, methanol (30 mL) was added and the mixture stirred overnight at room temperature. The crude product was purified by column chromatography.



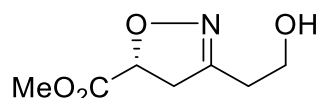
**3:** Yellow oil (0.41 g, 89% yield),  $R_f = 0.42$  (1:1 hexanes/ AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.89 (dd,  $J = 10.2, 7.9$  Hz, 1H,  $\text{OCHCO}$ ), 4.51–4.50 (m, 1H,  $\text{OCHO}$ ), 3.88–3.82 (m, 1H,  $\text{CH}_2\text{O}$ ), 3.75–3.71 (m, 1H,  $\text{CH}_2\text{O}$ ), 3.68 (s, 3H,  $\text{CH}_3$ ), 3.56–3.50 (m, 1H,  $\text{CH}_2\text{O}$ ), 3.42–3.39 (m, 1H,  $\text{CH}_2\text{O}$ ), 3.24–3.21 (m, 2H,  $\text{CHCH}_2\text{CH}$ ), 2.62–2.54 (m, 2H,  $\text{CH}_2\text{CH}_2\text{CH}$ ), 1.74–1.44 (m, 6H,  $\text{CH}_2\text{CH}_2\text{CH}_2$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 171.0, 156.9, 99.0, 98.9, 64.4, 64.3, 62.5, 62.4, 52.6, 41.6, 30.6, 27.9, 25.4, 19.6, 19.5; IR ( $\text{cm}^{-1}$ ): 3481, 2950, 2873, 2852, 2657, 1756, 1738, 1734, 1628, 1456,

1436, 1367, 1354, 1201, 1134, 1034, 869, 814, 752, 740; MS (ESI): calculated for C<sub>12</sub>H<sub>19</sub>NO<sub>5</sub> [M+H]<sup>+</sup> 258.1341, found 258.1340.

**[(5*R*)-3-(2-Hydroxyethyl)-2-isoxazolin-5-yl]carboxylic acid methyl ester (4)**

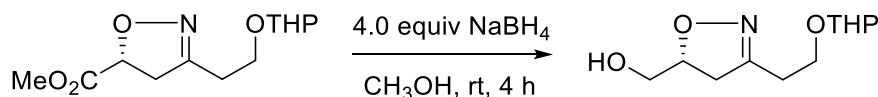


To a solution of **3** (276 mg, 1.07 mmol) in MeOH (8 mL) was added *p*-TsOH·H<sub>2</sub>O (37 mg, 0.21 mmol). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.

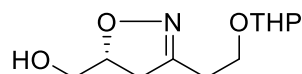


**4**: Yellow oil (176 mg, 95% yield), ee = 80%, R<sub>f</sub> = 0.42 (1:1 hexanes/ AcOEt). [α]<sub>D</sub><sup>20</sup> = -55.6 (c=1.28, CHCl<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 5.01–4.96 (m, 1H, OCHCO), 3.90–3.89 (m, 1H, HOCH<sub>2</sub>CH<sub>2</sub>), 3.78 (s, 1H, OCH<sub>3</sub>), 3.30–3.27 (m, 2H, NCCH<sub>2</sub>CH), 2.60–2.57 (m, 2H, OHCH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 170.9, 157.2, 59.2, 52.8, 41.7, 30.6; IR (cm<sup>-1</sup>): 3373, 2958, 2924, 2850, 1739, 1653, 1558, 1506, 1437, 1261, 1220, 1051, 1028, 873, 800, 501; MS (ESI): calculated for C<sub>7</sub>H<sub>11</sub>NO<sub>4</sub> [M+H]<sup>+</sup> 174.0766, found 174.0760; HPLC (Daicel AD-H column, *n*-hexane: *i*-PrOH = 80: 20, Flow rate = 1 mL/min, λ = 225 nm.): t<sub>major</sub> = 7.8 min, t<sub>minor</sub> = 9.1 min.

**(5*R*)-5-Hydroxymethyl-3-[2-(2-tetrahydropyranyloxy)ethyl]-2-isoxazoline (5)**



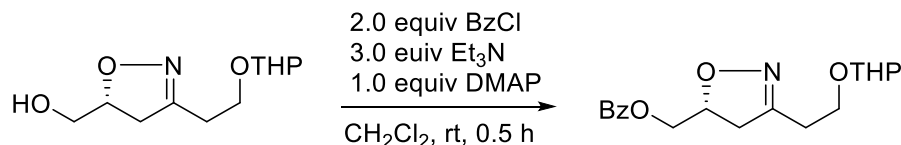
To a solution of **4** (244 mg, 0.95 mmol) in MeOH (10 mL) was added NaBH<sub>4</sub> (148 mg, 3.79 mmol). After stirring at room temperature for 4 h, the mixture was purified by column chromatography.



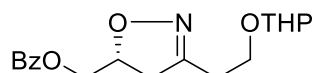
**5**: Colorless oil (206 mg, 95% yield), R<sub>f</sub> = 0.45 (AcOEt). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 4.64–4.63 (m, 1H, OCHCH<sub>2</sub>), 4.58 (m, 1H, OCHO), 3.95–3.47 (m, 6H, CH<sub>2</sub>O and CH<sub>2</sub>O and CH<sub>2</sub>O), 3.05–2.98 (m, 1H, CCH<sub>2</sub>CH), 2.91–2.85 (m, 1H, CCH<sub>2</sub>CH), 2.63–2.62 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>), 2.48 (m, 1H, CH<sub>2</sub>OH), 1.78–1.49 (m, 6H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 157.3, 98.6, 79.9, 64.1, 63.2, 62.1, 38.7, 30.3, 27.9, 25.0, 19.2; IR (cm<sup>-1</sup>): 3419, 2942, 2817, 2739, 2247, 1627, 1418, 1367, 1200, 1135, 1120, 905, 869, 814, 732; MS (ESI): calculated for C<sub>11</sub>H<sub>19</sub>NO<sub>4</sub> [M+H]<sup>+</sup> 230.1392, found

230.1383.

**(5*R*)-5-Benzoyloxymethyl-3-[2-(2-tetrahydropyranyloxy)ethyl]-2-isoxazoline (6)**

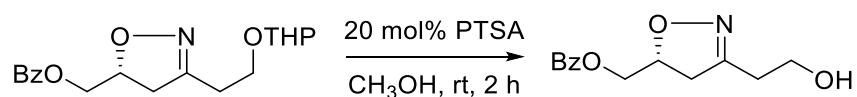


To a solution of **5** (414 mg, 1.81 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (20 mL) were added Et<sub>3</sub>N (760 μL, 5.43 mmol), DMAP (221 mg, 1.81 mmol) and benzoyl chloride (416 μL, 3.62 mmol). After stirring for 0.5 h at room temperature, the mixture was purified by column chromatography directly.

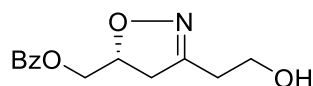


**6**: Colorless oil (585 mg, 97% yield), *R*<sub>f</sub> = 0.57 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 8.02–8.00 (m, 2H, ArH), 7.56–7.52 (m, 1H, ArH), 7.44–7.39 (m, 2H, ArH), 4.90–4.87 (m, 1H, OCHCH<sub>2</sub>), 4.57–4.50 (m, 1H, OCHO), 4.37–4.35 (m, 2H, BzOCH<sub>2</sub>), 3.94–3.44 (m, 4H, THPOCH<sub>2</sub> and CH<sub>2</sub>OCH), 3.20–3.12 (m, 1H, NCCH<sub>2</sub>CHO), 2.94–2.87 (m, 1H, NCCH<sub>2</sub>CHO), 2.72–2.58 (m, 2H, THPOCH<sub>2</sub>CH<sub>2</sub>), 1.76–1.48 (m, 6H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 166.2, 157.1, 157.0, 133.3, 129.9, 129.7, 129.5, 128.8, 128.6, 128.4, 98.9, 77.4, 77.1, 65.5, 64.4, 64.3, 62.5, 62.4, 39.9, 30.6, 29.7, 28.2, 28.1, 25.3, 19.6, 19.5; IR (cm<sup>-1</sup>): 3429, 3064, 3944, 1788, 1722, 1601, 1451, 1315, 1273, 1120, 1033, 974, 869, 814, 712; MS (ESI): calculated for C<sub>18</sub>H<sub>23</sub>NO<sub>5</sub> [M+H]<sup>+</sup> 334.1654, found 334.1643.

**(5*R*)-5-Benzoyloxymethyl-3-(2-hydroxyethyl)-2-isoxazoline (6')**



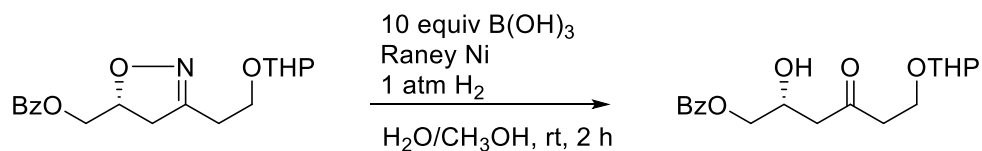
To a solution of **6** (294 mg, 0.88 mmol) in MeOH (4 mL) was added *p*-TsOH·H<sub>2</sub>O (30 mg, 0.2 equiv). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.



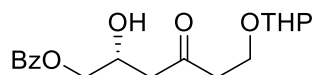
**6'**: Colorless oil (208 mg, 95% yield), *R*<sub>f</sub> = 0.24 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ: 7.94–7.92 (m, 2H, ArH), 7.48–7.44 (m, 1H, ArH), 7.35–7.32 (m, 2H, ArH), 4.81–4.80 (m, 1H, OCHCH<sub>2</sub>), 4.29–4.26 (m, 2H, BzOCH<sub>2</sub>), 3.78–3.74 (m, 2H, HOCH<sub>2</sub>), 3.41 (s, 1H, OH), 3.12–3.05 (m, 1H, NCCH<sub>2</sub>CHO), 2.84–2.78 (m, 1H, NCCH<sub>2</sub>CHO), 2.51–2.48 (m, 2H, HOCH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C

NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ : 166.0, 157.1, 132.9, 129.3, 129.2, 128.1, 65.2, 58.7, 39.5, 30.5; IR (cm<sup>-1</sup>): 3418, 3065, 2955, 2887, 1716, 1600, 1583, 1452, 1315, 1275, 1122, 1070, 871, 713; MS (ESI): calculated for C<sub>13</sub>H<sub>15</sub>NO<sub>4</sub> [M+H]<sup>+</sup> 250.1079, found 250.1076.

**(2R)-1-Benzoyloxy-2-hydroxy-6-(2-tetrahydropyranloxy)-4-hexanone (7)**

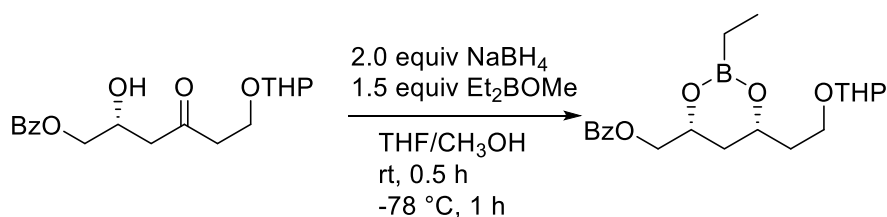


To a solution of **6** (208 mg, 0.62 mmol) in MeOH (30 mL) and H<sub>2</sub>O (6 mL) were added B(OH)<sub>3</sub> (385 mg, 6.22 mmol) and Raney nickel (250 mg). The reaction mixture was placed under 1 atm of H<sub>2</sub> and well stirred at room temperature for 2 h. The catalyst was filtered off and the filtrate concentrated. The residue was partitioned between sat. aq. NaHCO<sub>3</sub> solution (30 mL) and CH<sub>2</sub>Cl<sub>2</sub> (50 mL). The organic layer was separated. The aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 20 mL). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by column chromatography.



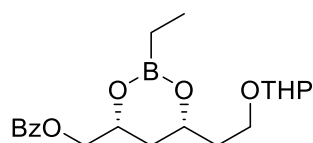
**7**: Colorless oil (177 mg, 85% yield), *R*<sub>f</sub> = 0.37 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ : 8.06–8.04 (m, 2H, ArH), 7.59–7.55 (m 1H, ArH), 7.46–7.42 (m, 2H, ArH), 4.58–4.57 (m, 1H, OCHO), 4.48–4.41 (m, 1H, CHOH), 4.39–4.35 (m, 2H, BzOCH<sub>2</sub>), 4.04–3.48 (m, 4H, THPOCH<sub>2</sub> and CH<sub>2</sub>OCH), 3.34 (m, 1H, CHOH), 2.80–2.73 (m, 4H, THPOCH<sub>2</sub>CH<sub>2</sub>COCH<sub>2</sub>), 1.77–1.50 (m, 6H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>)  $\delta$ : 209.1, 166.6, 133.2, 129.9, 129.7, 128.5, 99.1, 67.8, 66.1, 62.6, 62.5, 62.4, , 46.5, 43.7, 30.6, 29.7, 25.4, 19.6; IR (cm<sup>-1</sup>): 3459, 2944, 2875, 1717, 1451, 1315, 1275, 1027, 977, 869, 813, 713; MS (ESI): calculated for C<sub>18</sub>H<sub>24</sub>O<sub>6</sub> [M+H]<sup>+</sup> 337.1651, found 337.1649.

**(4R,6R)-6-Benzoyloxymethyl-2-ethyl-4-[2-(2-tetrahydropyranloxy)ethyl]-1,3,2-dioxaborolane (8)**



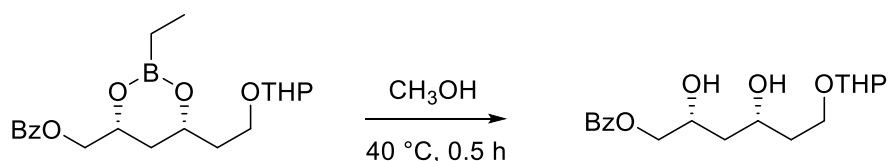
To a solution of **7** (1.73 g, 5.14 mmol) in THF (20 mL) and MeOH (4 mL) was added Et<sub>2</sub>BOMe

(7.71 mL, 7.71 mmol, 1 mol/L in THF), and the solution was stirred for 30 min at room temperature before being cooled to  $-78\text{ }^{\circ}\text{C}$ .  $\text{NaBH}_4$  (339 mg, 10.28 mmol) was added portionwise over 10 min (moderate gas evolution observed). After the solution was stirred for 1 h at  $-78\text{ }^{\circ}\text{C}$ , 20 mL of sat. aq.  $\text{NH}_4\text{Cl}$  was added to the cold solution. The mixture was allowed to warm to room temperature and then extracted with EtOAc (50 mL  $\times$  3). The combined layers were washed successively with saturated NaCl (20 mL) before being dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuo to give a viscous pale yellow oil. Flash chromatography with hexanes/AcOEt (5:1) as eluent provided the desired product.

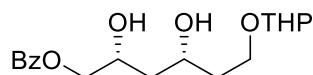


**8:** Colorless oil (1.86 g, 96% yield),  $R_f = 0.72$  (1:1 hexanes/AcOEt).  $^{11}\text{B}\{^1\text{H}\}$  NMR (128 MHz,  $\text{CDCl}_3$ )  $\delta$ : 31.46;  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.06–8.03 (m, 2H, ArH), 7.58–7.54 (m 1H, ArH), 7.46–7.42 (m, 2H, ArH), 4.60–4.57 (m, 1H, OCHO), 4.37–4.31 (m, 3H, CHOB and  $\text{BzOCH}_2$ ), 4.21–4.15 (m, 1H, CHOB), 3.99–3.46 (m, 4H, THPOCH $_2$  and  $\text{CH}_2\text{OCH}$ ), 2.06–1.76 (m, 4H, THPOCH $_2\text{CH}_2\text{COCH}_2$ ), 1.73–1.44 (m, 6H,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 0.89–0.85 (m, 3H,  $\text{BCH}_2\text{CH}_3$ ), 0.71–0.66 (m, 2H,  $\text{BCH}_2\text{CH}_3$ );  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$ : 166.3, 133.0, 129.9, 129.6, 128.3, 99.3, 98.6, 69.3, 68.1, 68.0, 67.6, 63.4, 63.1, 62.5, 62.1, 37.4, 37.3, 35.5, 35.4, 30.7, 25.4, 19.7, 19.5, 7.6; IR ( $\text{cm}^{-1}$ ): 2951, 2878, 1724, 1452, 1431, 1400, 1335, 1271, 1213, 1118, 1070, 1026, 981, 711; MS (ESI): calculated for  $\text{C}_{20}\text{H}_{29}\text{BO}_6$   $[\text{M}+\text{Na}]^+$  399.1955, found 399.1957.

**(2R,4R)-1-Benzoyloxy-6-(2-tetrahydropyranyloxy)hexane-2,4-diol (9)**



A solution of **8** (222 mg, 0.59 mmol) in MeOH (20 mL) was rotary evaporated at  $40\text{ }^{\circ}\text{C}$  for 4 times. The mixture was purified by column chromatography.

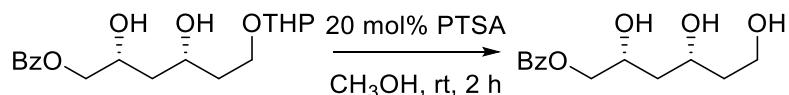


**9:** Colorless oil (198 mg, 99% yield),  $R_f = 0.24$  (AcOEt).  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.06–8.05 (m, 2H, ArH), 7.58–7.54 (m 1H, ArH), 7.45–7.41 (m, 2H, ArH), 4.59–4.55 (m, 1H, OCHO), 4.36–4.24 (m, 3H, CHOH and  $\text{BzOCH}_2$ ), 4.20–4.14 (m, 1H, CHOH), 4.01–3.49 (m, 5H, THPOCH $_2$  and

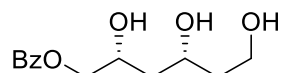


CH<sub>2</sub>OCH and CHOH), 1.87–1.72 (m, 4H, THPOCH<sub>2</sub>CH<sub>2</sub>COCH<sub>2</sub>), 1.61–1.52 (m, 6H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 166.6, 133.0, 129.9, 129.7, 128.3, 99.7, 99.2, 72.1, 71.1, 70.4, 70.3, 68.7, 66.1, 65.5, 63.3, 62.6, 39.6, 39.5, 36.9, 36.8, 30.7, 30.5, 25.2, 19.9, 19.6; IR (cm<sup>-1</sup>): 3435, 2872, 1720, 1452, 1383, 1352, 1315, 1275, 1118, 1072, 1026, 976, 713; MS (ESI): calculated for C<sub>18</sub>H<sub>26</sub>O<sub>6</sub> [M+Na]<sup>+</sup> 361.1627, found 361.1624.

**(2R,4R)-1-Benzoyloxyhexane-2,4,6-triol (10)**

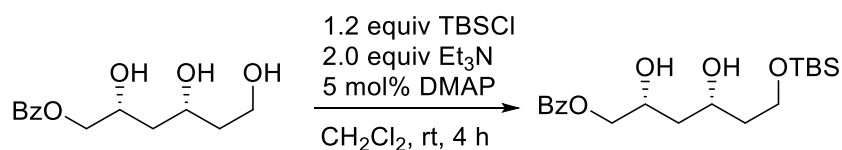


To a solution of **9** (199 mg, 0.59 mmol) in MeOH (8 mL) was added *p*-TsOH·H<sub>2</sub>O (21 mg, 0.12 mmol). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.

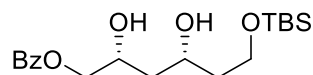


**10**: Colorless oil (134 mg, 89% yield), R<sub>f</sub> = 0.24 (AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ: 8.06–8.04 (m, 2H, ArH), 7.59–7.55 (m 1H, ArH), 7.46–7.42 (m, 2H, ArH), 4.37–4.18 (m, 4H, CH<sub>2</sub>OH and BzOCH<sub>2</sub>), 4.07 (s, 1H, CH<sub>2</sub>OH), 3.93–3.79 (m, 3H, CHOH and CHOH and OH), 2.75 (s, 1H, OH), 1.80–1.66 (m, 4H, CH<sub>2</sub>CHOHCH<sub>2</sub>CHOH); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 166.8, 133.2, 129.7, 129.6, 128.4, 72.2, 70.8, 68.9, 61.3, 39.4, 38.7, 29.7; IR (cm<sup>-1</sup>): 3375, 2918, 1716, 1452, 1279, 1116, 1099, 1070, 711; MS (ESI): calculated for C<sub>13</sub>H<sub>18</sub>O<sub>5</sub> [M+Na]<sup>+</sup> 277.1052, found 277.1048.

**(2R,4R)-1-Benzoyloxy-6-(tert-butyldimethylsilyloxy)hexane-2,4-diol (11)**

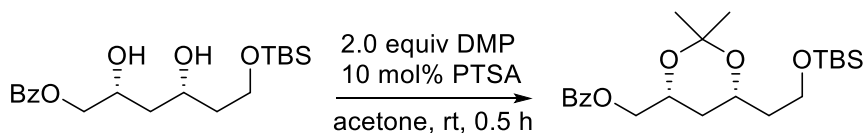


To a stirred solution of **10** (117 mg, 0.46 mmol), Et<sub>3</sub>N (129 μL, 0.92 mmol) and DMAP (3 mg, 0.023 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (4 mL) was added TBS-Cl (83 mg, 0.552 mmol). After stirring for 4 h at room temperature, the mixture was poured into sat. aq. NH<sub>4</sub>Cl (25 mL). The organic layer was separated, and the aqueous portion extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL). The combined organic extracts were washed with sat. aq. NaCl (20 mL) and dried. Removal of the solvent in vacuo followed by column chromatography of the residue afforded pure **11**.

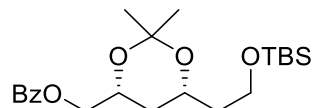


**11:** Colorless oil (147 mg, 87% yield),  $R_f = 0.47$  (1:1 hexanes/AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.07–8.05 (m, 2H, ArH), 7.58–7.54 (m 1H, ArH), 7.45–7.41 (m, 2H, ArH), 4.32–4.17 (m, 5H,  $\text{CH}_2\text{OTBS}$  and OH and  $\text{BzOCH}_2$ ), 4.05 (s, 1H,  $\text{CHOH}$ ), 3.94–3.81 (m, 2H,  $\text{CHOH}$  and  $\text{CHOH}$ ), 1.77–1.66 (m, 4H,  $\text{CH}_2\text{CHOHCH}_2\text{CHOH}$ ), 0.89 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 0.08 (s, 6H,  $\text{Si}(\text{CH}_3)_2$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 166.6, 133.0, 130.0, 129.7, 128.3, 72.9, 70.3, 68.7, 62.6, 25.8, 18.1, –5.6, –5.6; IR ( $\text{cm}^{-1}$ ): 3416, 2953, 2829, 2856, 1722, 1452, 1275, 1257, 1097, 1072, 837, 777, 711; MS (ESI): calculated for  $\text{C}_{19}\text{H}_{32}\text{O}_5\text{Si}$   $[\text{M}+\text{Na}]^+$  391.1917, found 391.1913.

**(4*R*,6*R*)-6-Benzoyloxymethyl-4-[2-(*tert*-butyldimethylsilyloxy)ethyl]-2,2-dimethyl-1,3-dioxane (12)**

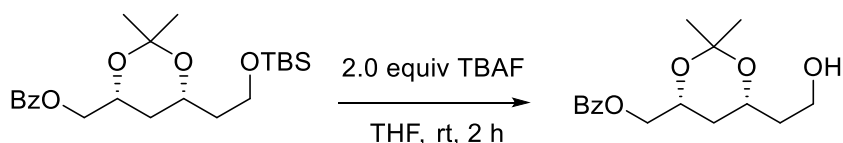


To a solution of **11** (111 mg, 0.30 mmol) in acetone (4 mL) were added 2,2-dimethoxypropane (63 mg, 0.60 mmol) and PTSA (5 mg, 0.03 mmol). The resulting mixture was stirred at room temperature until TLC analysis indicated consumption of the starting material (0.5 h).  $\text{Et}_3\text{N}$  (3 mL) was added, and the mixture concentrated and purified by column chromatography.

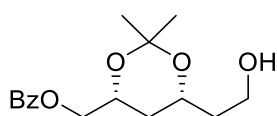


**12:** Colorless oil (121 mg, 99% yield),  $R_f = 0.66$  (3:1 hexanes/AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.06–8.03 (m, 2H, ArH), 7.55–7.53 (m 1H, ArH), 7.45–7.41 (m, 2H, ArH), 4.30–4.09 (m, 4H,  $\text{CH}_2\text{OTBS}$  and  $\text{BzOCH}_2$ ), 3.75–3.65 (m, 2H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$ ), 1.69–1.58 (m, 3H,  $\text{CH}_2\text{CHOCH}_2\text{CHO}$ ), 1.58 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.47 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.42–1.33 (m, 1H, one of  $\text{CH}_2\text{CHOCCH}_2\text{CHOC}$ ), 0.89 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 0.04 (s, 6H,  $\text{Si}(\text{CH}_3)_2$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 166.3, 132.9, 130.0, 129.6, 128.3, 98.7, 67.5, 67.4, 65.2, 58.6, 39.4, 33.6, 30.0, 25.9, 19.7, 18.2, –5.4; IR ( $\text{cm}^{-1}$ ): 2953, 2928, 1724, 1471, 1379, 1273, 1170, 1105, 1099, 835, 775, 711; MS (ESI): calculated for  $\text{C}_{22}\text{H}_{36}\text{O}_5\text{Si}$   $[\text{M}+\text{Na}]^+$  431.2230, found 431.2222.

**(4*R*,6*R*)-6-Benzoyloxymethyl-2,2-dimethyl-4-(2-hydroxyethyl)-1,3-dioxane (13)**

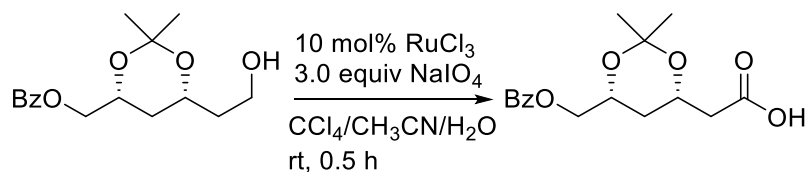


To a stirred solution of **12** (119 mg, 0.29 mmol) in THF (4 mL) was added 1 M tetrabutylammonium fluoride in THF (0.58 mL, 0.58 mmol). The mixture was stirred for 2 h at room temperature. The reaction was quenched with aqueous  $\text{NH}_4\text{Cl}$ . The layers were separated, and the aqueous layer extracted with AcOEt. The combined organic layers were washed with brine and dried over  $\text{Na}_2\text{SO}_4$ . After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography.

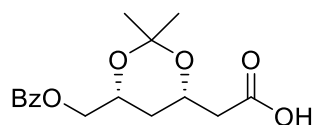


**13**: Colorless oil (84 mg, 99% yield),  $R_f = 0.41$  (1:1 hexanes/AcOEt).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.05–8.03 (m, 2H, ArH), 7.57–7.54 (m 1H, ArH), 7.45–7.41 (m, 2H, ArH), 4.29–4.15 (m, 4H,  $\text{CH}_2\text{OH}$  and  $\text{BzOCH}_2$ ), 3.78–3.75 (m, 2H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$ ), 2.56 (s, 1H, OH), 1.78–1.72 (m, 2H,  $\text{CH}_2\text{CHOCH}_2\text{CHO}$ ), 1.58–1.55 (m, 1H, one of  $\text{CH}_2\text{CHOCH}_2\text{CHO}$ ), 1.48 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.47–1.43 (m, 1H, one of  $\text{CH}_2\text{CHOCH}_2\text{CHO}$ ), 1.42 (s, 3H,  $\text{C}(\text{CH}_3)_2$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 166.4, 133.0, 129.9, 129.6, 128.3, 98.87, 68.5, 67.3, 62.3, 60.4, 38.2, 33.0, 29.9, 29.6, 19.7; IR ( $\text{cm}^{-1}$ ): 3487, 2920, 1720, 1452, 1381, 1276, 1201, 1168, 1114, 912, 732, 713, 648; MS (ESI): calculated for  $\text{C}_{16}\text{H}_{22}\text{O}_5$   $[\text{M}+\text{Na}]^+$  317.1365, found 317.1358.

**[(4*S*,6*R*)-6-Benzoyloxymethyl-2,2-dimethyl-1,3-dioxan-4-yl]acetic acid (**14**)**

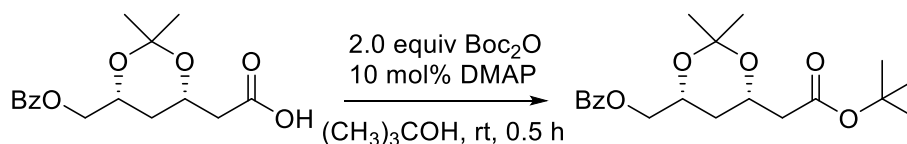


To a stirred solution of **13** (152 mg, 0.52 mmol) and  $\text{NaIO}_4$  (334 mg, 1.56 mmol) in  $\text{CCl}_4$  (2 mL),  $\text{CH}_3\text{CN}$  (2 mL), and  $\text{H}_2\text{O}$  (1.2 mL) was added ruthenium trichloride hydrate (12 mg, 0.052 mmol), and the mixture stirred for 30 min at room temperature. After this period,  $\text{CH}_2\text{Cl}_2$  (25 mL) was added, and the layers were separated. The aqueous layer was extracted with  $\text{CH}_2\text{Cl}_2$ . The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$ . The mixture was concentrated and purified by column chromatography.

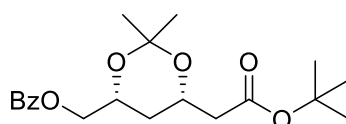


**14:** Colorless oil (138 mg, 86% yield),  $R_f = 0.40$  (1:1 hexanes/AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.05–8.03 (m, 2H, ArH), 7.57–7.53 (m 1H, ArH), 7.45–7.41 (m, 2H, ArH), 4.39–4.25 (m, 4H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{BzOCH}_2$ ), 2.63–2.45 (m, 2H,  $\text{CH}_2\text{COOH}$ ), 1.71–1.68 (m, 1H,  $\text{OCHCH}_2\text{CHO}$ ), 1.48 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.43–1.38 (m, 1H,  $\text{OCHCH}_2\text{CHO}$ ), 1.41 (s, 3H,  $\text{C}(\text{CH}_3)_2$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 176.2, 166.4, 133.0, 129.8, 129.6, 128.3, 99.2, 67.2, 67.1, 65.3, 41.0, 32.6, 29.7, 19.5; IR ( $\text{cm}^{-1}$ ): 3200, 2993, 2945, 1716, 1383, 1276, 1201, 1168, 1118, 995, 958, 947, 713; MS (ESI): calculated for  $\text{C}_{16}\text{H}_{20}\text{O}_6$   $[\text{M}+\text{Na}]^+$  331.1158, found 331.1158.

**[(4*S*,6*R*)-6-Benzoyloxymethyl-2,2-dimethyl-1,3-dioxan-4-yl]acetic acid *t*-butyl ester (**15**)**



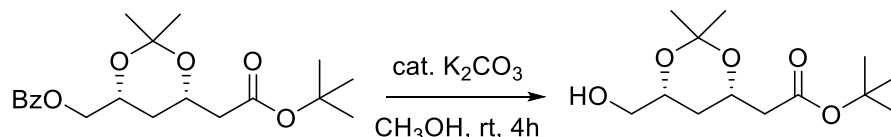
To a stirred solution of **14** (115 mg, 0.37 mmol) in *tert*-butyl alcohol (4 mL) were added BOC anhydride (162 mg, 0.74 mmol), DMAP (5 mg, 0.037 mmol), and the reaction mixture was stirred for 0.5 h at room temperature. The reaction was quenched with aqueous  $\text{NH}_4\text{Cl}$ . The layers were separated, and the aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine and dried over  $\text{Na}_2\text{SO}_4$ . After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography.



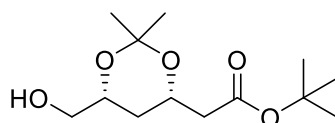
**15:** Colorless oil (116 mg, 86% yield),  $ee = 74\%$ ,  $R_f = 0.37$  (5:1 hexanes/AcOEt).  $[\alpha]_D^{20} = -4.5$  ( $c=0.88$ ,  $\text{CHCl}_3$ ).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.04–8.02 (m, 2H, ArH), 7.56–7.52 (m 1H, ArH), 7.44–7.40 (m, 2H, ArH), 4.32–4.23 (m, 4H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{BzOCH}_2$ ), 2.47–2.30 (m, 2H,  $\text{CH}_2\text{COOC}(\text{CH}_3)_3$ ), 1.67–1.63 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ), 1.47 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.42 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 1.39 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.37–1.31 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 169.9, 166.3, 132.9, 129.3, 129.6, 128.3, 98.9, 80.6, 67.3, 67.2, 65.8, 42.6, 32.8, 29.9, 27.9, 19.6; IR ( $\text{cm}^{-1}$ ): 2980, 2941, 1724, 1602, 1452, 1381, 1315, 1275, 1153, 1114, 1070, 995, 844, 713; MS (ESI): calculated for  $\text{C}_{20}\text{H}_{28}\text{O}_6$   $[\text{M}+\text{Na}]^+$  387.1784, found 387.1782; HPLC

(Daicel OD-H column, *n*-hexane: *i*-PrOH = 90: 10, Flow rate = 0.5 mL/min,  $\lambda$  = 254 nm.):  $t_{\text{major}}$  = 13.7 min,  $t_{\text{minor}}$  = 10.9 min.

**[(4*S*,6*R*)-2,2-Dimethyl-6-hydroxymethyl-1,3-dioxan-4-yl]acetic acid *t*-butyl ester (16)**

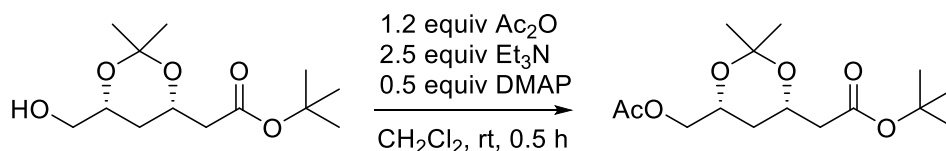


A mixture of **15** (98 mg, 0.25 mmol), solid  $\text{K}_2\text{CO}_3$  (13 mg, 0.125 mmol) and MeOH (8 mL) was stirred at room temperature for 4 h. It was concentrated and purified by column chromatography.

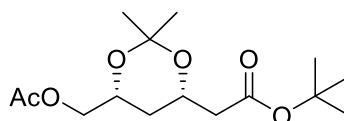


**16**: Colorless oil (57 mg, 87% yield),  $R_f$  = 0.42 (1:1 hexanes/AcOEt).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.29–4.00 (m, 1H, one of  $\text{CH}_2\text{OH}$ ), 3.99–3.96 (m, 1H, one of  $\text{CH}_2\text{OH}$ ), 3.59–3.45 (m, 2H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$ ), 2.44–2.26 (m, 3H,  $\text{CH}_2\text{COOC}(\text{CH}_3)_3$  and  $\text{CH}_2\text{OH}$ ), 1.48–1.28 (m, 2H,  $\text{OCHCH}_2\text{CHO}$ ), 1.44 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.42 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 1.36 (s, 3H,  $\text{C}(\text{CH}_3)_2$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 170.1, 98.8, 80.6, 69.5, 65.8, 65.7, 42.6, 31.8, 29.9, 28.0, 19.7; IR ( $\text{cm}^{-1}$ ): 3449, 2980, 2937, 1730, 1369, 1315, 1259, 1201, 1153, 1078, 1024, 950, 842; MS (ESI): calculated for  $\text{C}_{13}\text{H}_{24}\text{O}_5$   $[\text{M}+\text{Na}]^+$  283.1521, found 283.1516.

**[(4*S*,6*R*)-6-Acetoxyethyl-2,2-dimethyl-1,3-dioxan-4-yl]acetic acid *t*-butyl ester (17)**



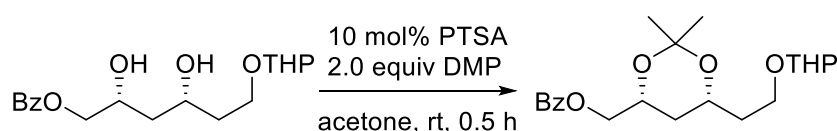
To a solution of **16** (47 mg, 0.18 mmol) in  $\text{CH}_2\text{Cl}_2$  (5 mL) were added  $\text{Et}_3\text{N}$  (63  $\mu\text{L}$ , 0.45 mmol), DMAP (11 mg, 0.09 mmol) and  $\text{Ac}_2\text{O}$  (23 mg, 0.22 mmol). After stirring for 0.5 h at room temperature, the mixture was purified by column chromatography directly.



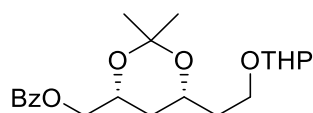
**17**: Colorless oil (54 mg, 99% yield),  $R_f$  = 0.78 (1:1 hexanes/AcOEt).  $[\alpha]_{\text{D}}^{20}$  =  $-11.67$  ( $c=0.54$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.30–3.97 (m, 4H,  $\text{AcOCH}_2$  and  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$ ), 2.46–2.83 (m, 2H,  $\text{CH}_2\text{COOC}(\text{CH}_3)_3$ ), 2.07 (s, 3H,  $\text{OCOCH}_3$ ), 1.58–1.54 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ), 1.45 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.44 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 1.38 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.29–

1.21 (m, 1H, one of OCHCH<sub>2</sub>CHO); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ: 170.9, 170.1, 98.9, 80.7, 67.2, 67.0, 65.8, 42.6, 32.5, 29.9, 28.1, 20.9, 19.6; IR (cm<sup>-1</sup>): 2980, 1739, 1367, 1238, 1155, 1035, 864; MS (ESI): calculated for C<sub>15</sub>H<sub>26</sub>O<sub>6</sub> [M+Na]<sup>+</sup> 325.1627, found 325.1619.

**(4*R*,6*R*)-6-Benzoyloxymethyl-2,2-dimethyl-4-[2-(2-tetrahydropyranyloxy)ethyl]-1,3-dioxane (18)**

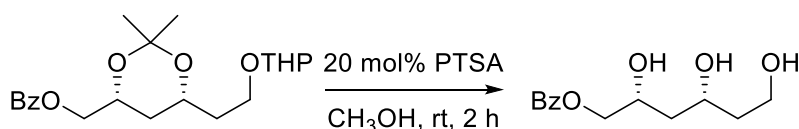


To a solution of **9** (113 mg, 0.33 mmol) in acetone (4 mL) were added 2,2-dimethoxypropane (69 mg, 0.66 mmol) and PTSA (6 mg, 0.033 mmol). The resulting mixture was stirred at room temperature until TLC analysis indicated consumption of the starting material (0.5 h). Et<sub>3</sub>N (3 mL) was added, and the mixture concentrated and purified by column chromatography.

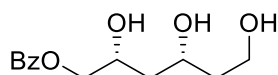


**18**: Colorless oil (123 mg, 99% yield), *R*<sub>f</sub> = 0.77 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ: 8.05–8.04 (m, 2H, ArH), 7.56–7.54 (m 1H, ArH), 7.44–7.42 (m, 2H, ArH), 4.59–4.55 (m, 1H, OCHO), 4.31–4.25 (m, 3H, CHOC(CH<sub>3</sub>)<sub>2</sub> and BzOCH<sub>2</sub>), 4.09–4.07 (m, 1H, CHOC(CH<sub>3</sub>)<sub>2</sub>), 3.86–3.43 (m, 4H, THPOCH<sub>2</sub> and CH<sub>2</sub>OCH), 1.82–1.46 (m, 10H, THPOCH<sub>2</sub>CH<sub>2</sub>COCH<sub>2</sub> and CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 166.4, 132.9, 130.1, 129.6, 128.3, 99.3, 98.8, 98.5, 99.3, 98.8, 98.5, 67.5, 67.4, 66.7, 65.5, 63.2, 61.9, 36.5, 36.4, 33.6, 33.4, 30.7, 30.6, 30.0, 29.6, 25.4, 19.7, 19.4; IR (cm<sup>-1</sup>): 2941, 2370, 1722, 1381, 1274, 1201, 1118, 1026, 867, 711, 412; MS (ESI): calculated for C<sub>21</sub>H<sub>30</sub>O<sub>6</sub> [M+Na]<sup>+</sup> 401.1940, found 401.1937.

**(2*R*,4*R*)-1-Benzoyloxyhexane-2,4,6-triol (10)**

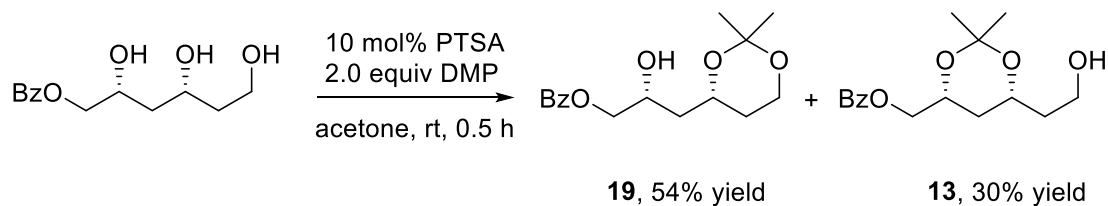


To a solution of **18** (223 mg, 0.59 mmol) in MeOH (4 mL) was added *p*-TsOH·H<sub>2</sub>O (21 mg, 0.12 mmol). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.

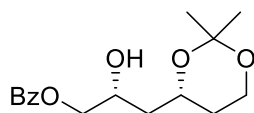


**10**: Colorless oil (134 mg, 89% yield),  $R_f = 0.24$  (AcOEt).

**(2R)-1-Benzoyloxy-3-[(4R)-2,2-dimethyl-1,3-dioxan-4-yl]-2-propanol (19)**

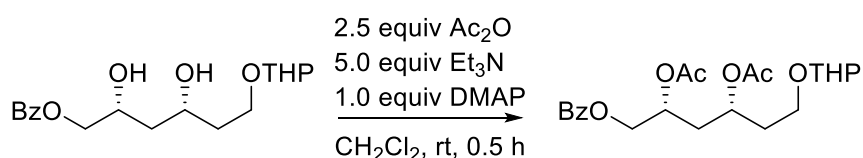


To a solution of **10** (168 mg, 0.66 mmol) in acetone (4 mL) were added 2,2-dimethoxypropane (138 mg, 1.32 mmol) and PTSA (11 mg, 0.066 mmol). The resulting mixture was stirred at room temperature until TLC analysis indicated consumption of the starting material (0.5 h). Et<sub>3</sub>N (3 mL) was added, and the mixture concentrated and purified by column chromatography.

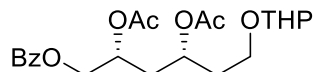


**19**: Colorless oil (106 mg, 54% yield),  $R_f = 0.44$  (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$ : 8.03–8.02 (m, 2H, ArH), 7.55–7.54 (m 1H, ArH), 7.43–7.41 (m, 2H, ArH), 4.29–4.23 (m, 3H, CHOH and BzOCH<sub>2</sub>), 4.16–4.12 (m, 1H, CHOC(CH<sub>3</sub>)<sub>2</sub>), 3.81–3.70 (m, 2H, CH<sub>2</sub>OC(CH<sub>3</sub>)<sub>2</sub>), 2.57 (s, 1H, OH), 1.78–1.41 (m, 4H, OCH<sub>2</sub>CH<sub>2</sub>CHOCH<sub>2</sub>), 1.47 (s, 3H, C(CH<sub>3</sub>)<sub>2</sub>), 1.41 (s, 3H, C(CH<sub>3</sub>)<sub>2</sub>); MS (ESI): calculated for C<sub>16</sub>H<sub>22</sub>O<sub>5</sub> [M+Na]<sup>+</sup> 317.1365, found 317.1358.

**(2R,4R)-1-Benzoyloxy-2,4-diacetoxy-6-(2-tetrahydropyranyloxy)hexane (20)**



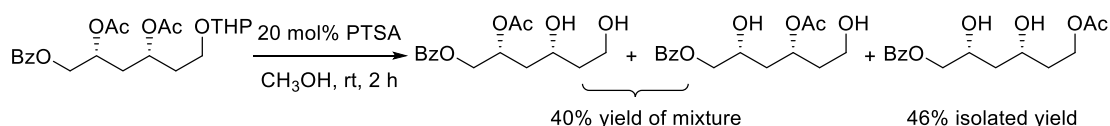
To a solution of **9** (100 mg, 0.30 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (5 mL) were added Et<sub>3</sub>N (210  $\mu$ L, 1.5 mmol), DMAP (37 mg, 0.3 mmol) and Ac<sub>2</sub>O (77 mg, 0.75 mmol). After stirring for 0.5 h at room temperature, the mixture was purified by column chromatography directly.



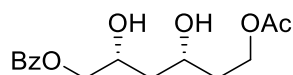
**20**: Colorless oil (125 mg, 99% yield),  $R_f = 0.81$  (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$ : 8.01–7.99 (m, 2H, ArH), 7.56–7.53 (m 1H, ArH), 7.44–7.40 (m, 2H, ArH), 5.32–5.12 (m, 2H, two CHOCOCH<sub>3</sub>), 4.54–4.46 (m, 2H, OCHO and one of BzOCH<sub>2</sub>), 4.33–4.28 (m, 1H, one of BzOCH<sub>2</sub>), 3.83–3.73 (m, 2H, THPOCH<sub>2</sub>), 3.47–3.33 (m, 2H, CH<sub>2</sub>OCH), 2.07–2.03 (m, 8H, two of THPOCH<sub>2</sub>CH<sub>2</sub>COCH<sub>3</sub> and two COCH<sub>3</sub>), 1.92–1.86 (m, 2H, two of THPOCH<sub>2</sub>CH<sub>2</sub>COCH<sub>2</sub>), 1.79–

1.45 (m, 6H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ: 170.4, 170.2, 166.0, 133.1, 129.7, 128.4, 99.1, 98.5, 68.7, 68.6, 68.5, 65.2, 63.4, 63.3, 62.3, 61.9, 35.1, 33.9, 33.8, 30.5, 29.6, 25.3, 21.1, 20.9, 19.5, 19.2; IR (cm<sup>-1</sup>): 2943, 2872, 2850, 1739, 1452, 1373, 1276, 1236, 1120, 1070, 1026, 906, 732, 713; MS (ESI): calculated for C<sub>22</sub>H<sub>30</sub>O<sub>8</sub> [M+Na]<sup>+</sup> 445.1838, found 445.1835.

**(2R,4R)-6-Acetoxy-1-benzoyloxyhexane-2,4-diol (21)**

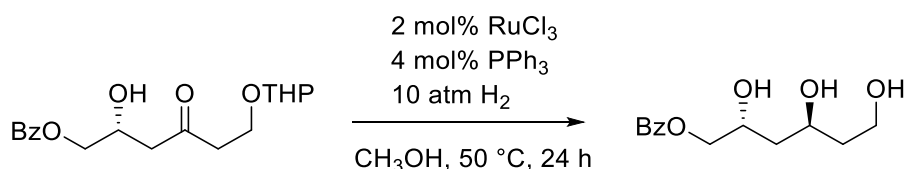


To a solution of **20** (196 mg, 0.46 mmol) in MeOH (4 mL) was added *p*-TsOH·H<sub>2</sub>O (16 mg, 0.09 mmol). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.

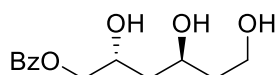


**21**: Colorless oil (62 mg, 46% yield), R<sub>f</sub> = 0.37 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ: 8.05–8.02(m, 2H, ArH), 7.57–7.55 (m 1H, ArH), 7.45–7.42 (m, 2H, ArH), 4.37–4.23 (m, 4H, CH<sub>2</sub>OAc and BzOCH<sub>2</sub>), 4.14–4.10 (m, 1H, CHOH), 4.00–3.96 (m, 1H, CHOH), 3.77 (s, 1H, OH), 3.68 (s, 1H, OH), 2.05 (s, 3H, CH<sub>3</sub>CO), 1.85–1.69 (m, 4H, CH<sub>2</sub>CHOHCH<sub>2</sub>CHOH); MS (ESI): calculated for C<sub>15</sub>H<sub>20</sub>O<sub>6</sub> [M+Na]<sup>+</sup> 319.1158, found 319.1153.

**(2R,4S)-1-Benzoyloxyhexane-2,4,6-triol**



**7** (117.6 mg, 0.35 mmol) in degassed methanol (5 mL) was cannulated to a Schlenk tube containing a degassed mixture of RuCl<sub>3</sub> (1.5 mg, 0.007 mmol) and PPh<sub>3</sub> (3.6 mg, 0.014 mmol). The Schlenk tube was placed in a glove box and the reaction mixture transferred to a stainless steel autoclave, which was purged with hydrogen and pressurized to 10 bar. The autoclave was heated to 50 °C. After stirring for 24 h, the autoclave was cooled to room temperature, hydrogen was vented, and the mixture was concentrated and purified by column chromatography.



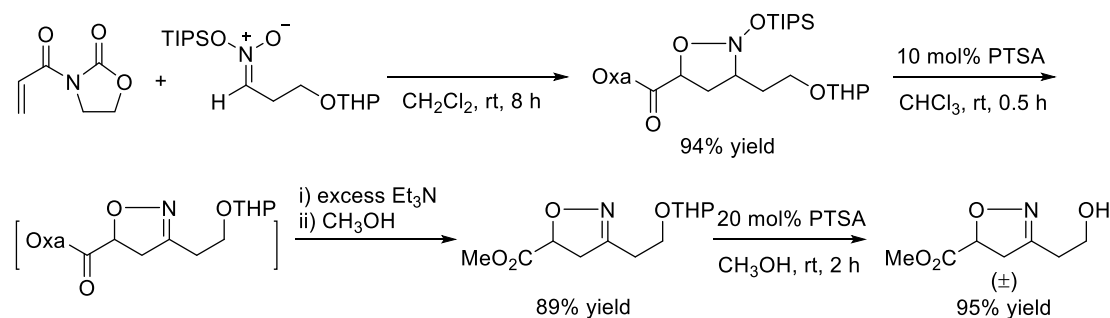
Colorless oil (72 mg, 81% yield), R<sub>f</sub> = 0.24 (AcOEt). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 8.04–8.03 (m,



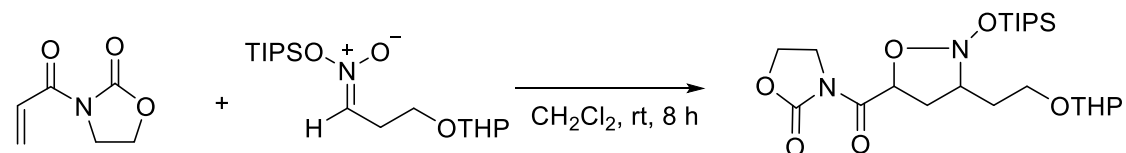
2H, ArH), 7.59–7.55 (m, 1H, ArH), 7.46–7.42 (m, 2H, ArH), 4.36–4.19 (m, 4H, CH<sub>2</sub>OH and BzOCH<sub>2</sub>), 4.08 (s, 1H, CH<sub>2</sub>OH), 3.91–3.81 (m, 2H, CHOH and CHOH), 2.78 (s, 1H, OH), 1.78–1.66 (m, 4H, CH<sub>2</sub>CHOHCH<sub>2</sub>CHOH); MS (ESI): calculated for C<sub>13</sub>H<sub>18</sub>O<sub>5</sub> [M+Na]<sup>+</sup> 277.1052, found 277.1048.

## 2. Preparation of the racemic samples of compounds 4 and 15

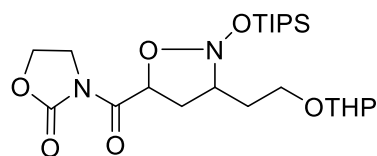
### Preparation of the racemic sample of compound 4



### *N*-{(3*S*\*,5*R*\*)-[3-(2-(2-Tetrahydropyranyloxy)ethyl)-2-triisopropylsilyloxy-5-isoxazolidinyl]carbonyl}-1,3-oxazolidin-2-one



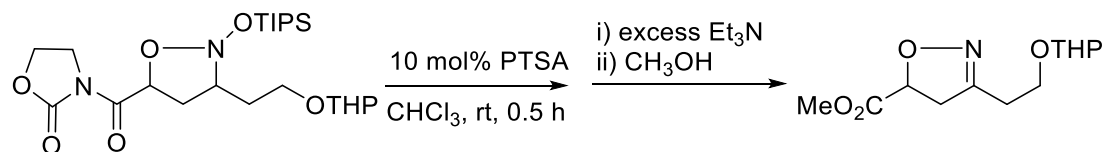
To a dry Schlenk tube were added *N*-acryloyl-1,3-oxazolidin-2-one (282 mg, 2 mmol) and the silyl nitronate (3.0 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (3 mL). The mixture was stirred at room temperature overnight and monitored by TLC. After the reaction was completed, the product was purified by silica gel chromatography.



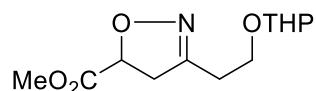
Yellow oil (914 mg, 94 % yield), *R*<sub>f</sub> = 0.40 (1:1 hexanes/AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ: 5.81–5.74 (m, 1H, CH<sub>2</sub>CHO), 4.56–4.55 (m, 1H, OCHO), 4.50–4.42 (m, 2H, CH<sub>2</sub>O), 4.14–3.99 (m, 2H, CH<sub>2</sub>O), 3.85–3.77 (m, 2H, OCH<sub>2</sub>CH<sub>2</sub>), 3.54–3.40 (m, 3H, NCH and NCH<sub>2</sub>), 2.77–2.69 (m, 1H, CHCH<sub>2</sub>CH), 2.35–2.29 (m, 1H, CHCH<sub>2</sub>CH), 2.18–2.14 (m, 1H, CH<sub>2</sub>CH<sub>2</sub>), 1.89–1.49 (m, 7H, CH<sub>2</sub>CH<sub>2</sub> and CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 1.27–1.16 (m, 3H, SiCH), 1.10–1.05 (m, 18H, SiCH(CH<sub>3</sub>)<sub>2</sub>); MS (ESI):

calculated for C<sub>23</sub>H<sub>42</sub>N<sub>2</sub>O<sub>7</sub>Si [M+Na]<sup>+</sup> 509.2659, found 509.2659.

**{{(5*R*\*)-[3-(2-(2-Tetrahydropyranloxy)ethyl)]-2-isoxazolin-5-yl}carboxylic acid methyl ester**

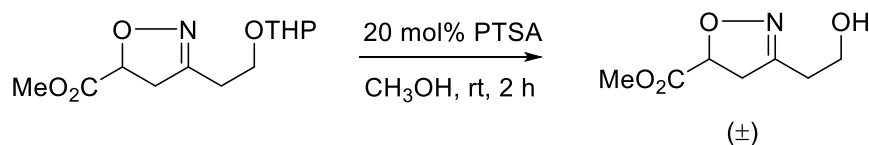


To a solution of the starting isoxazolidine (0.86 g, 1.78 mmol) in CHCl<sub>3</sub> (15 mL) was added PTSA (31 mg, 0.178 mmol) at 0 °C. The mixture was allowed to warm to room temperature and stirred until complete consumption of the starting material (0.5 h). Vacuum was applied to remove the solvent before Et<sub>3</sub>N (5 mL) was added. After stirring for 5 min, methanol (30 mL) was added and the mixture stirred overnight at room temperature. The crude product was purified by column chromatography.

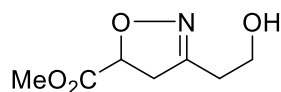


Yellow oil (0.41 g, 89% yield), R<sub>f</sub> = 0.42 (1:1 hexanes/ AcOEt).

**(±)-[3-(2-Hydroxyethyl)-2-isoxazolin-5-yl]carboxylic acid methyl ester**

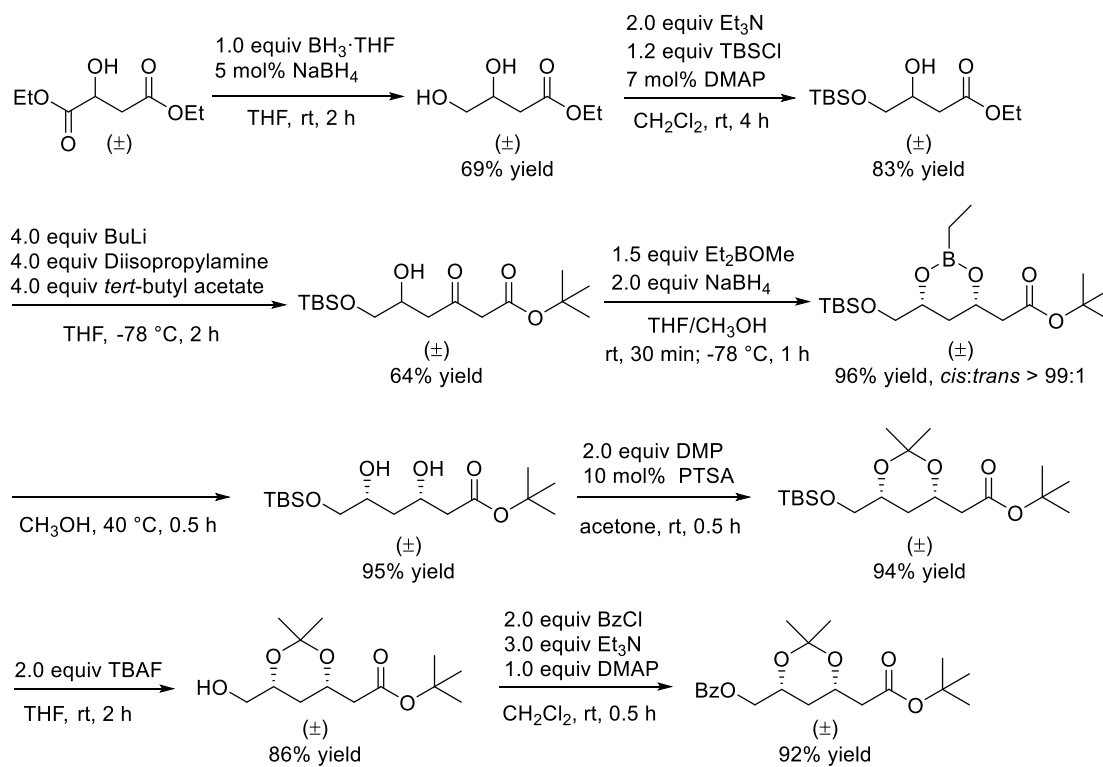


To a solution of the starting 2-isoxazoline (276 mg, 1.07 mmol) in MeOH (8 mL) was added *p*-TsOH·H<sub>2</sub>O (37 mg, 0.21 mmol). After stirring for 2 h at room temperature, the mixture was concentrated and purified by column chromatography.

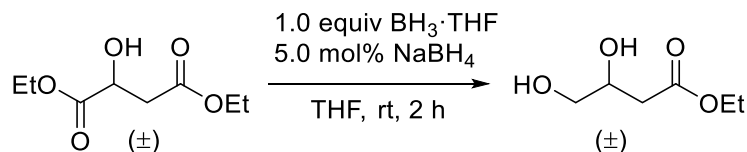


Yellow oil (176 mg, 95% yield), R<sub>f</sub> = 0.42 (1:1 hexanes/ AcOEt).

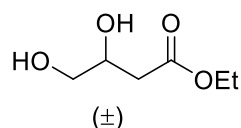
**Preparation of the racemic sample of compound 15**



#### (±)-3,4-Dihydroxybutanoic acid ethyl ester



To a solution of diethyl DL-malate (2 g, 10.5 mmol) in THF (10 mL) was added dropwise  $\text{BH}_3$  in THF (10.6 mL, 10.6 mmol) over 30 min at  $0^\circ\text{C}$  under  $\text{N}_2$ .  $\text{NaBH}_4$  powder (20 mg, 0.525 mmol) was added in one portion (exothermic) under vigorous stirring at that temperature until evolution of hydrogen ceased (30 min). Then, the mixture was allowed to warm to room temperature and stirred until TLC analysis indicated consumption of the starting material (2 h). To the reaction mixture were added MeOH (10 mL) and PTSA (0.525 mg, 5 mol %) and the resulting slightly cloudy solution was stirred for 30 min at room temperature, followed by concentration to give a colorless gum. This was dissolved in MeOH (10 mL) and the resulting solution was concentrated via rotary evaporation again. This operation was repeated. To the residue was added benzene (10 mL) and the solution concentrated via rotary evaporation. This was repeated to eliminate MeOH and  $\text{B}(\text{OMe})_3$  as thoroughly as possible to give a clear, colorless gum.

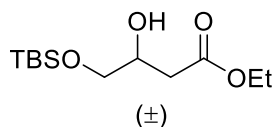


Colorless oil (1.07 g, 69% yield),  $R_f = 0.42$  (AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.20–4.19 (m, 1H,  $\text{CH}_2\text{OH}$ ), 4.06–3.94 (m, 4H,  $\text{CHOH}$  and  $\text{CH}_3\text{CH}_2\text{O}$  and  $\text{OH}$ ), 3.53–3.35 (m, 2H,  $\text{CH}_2\text{OH}$ ), 2.38–2.36 (m, 2H,  $\text{CHOHCH}_2\text{CO}$ ), 1.14 (t,  $J = 7.1$  Hz, 3H,  $\text{OCH}_2\text{CH}_3$ ).

**(±)-4-(*tert*-Butyldimethylsilyloxy)-3-hydroxybutanoic acid ethyl ester**

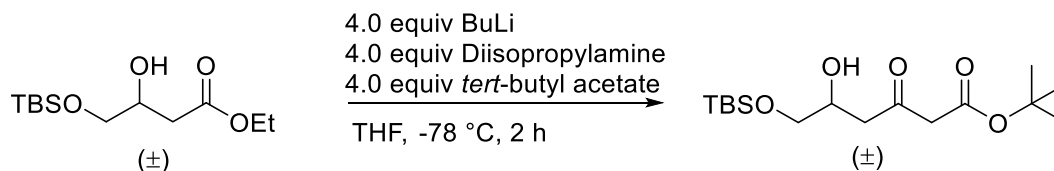


To a stirred solution of the starting dihydroxy ester (2.79 g, 18 mmol),  $\text{Et}_3\text{N}$  (5 mL, 0.36 mmol) and DMAP (146.4 mg, 1.21 mmol) in  $\text{CH}_2\text{Cl}_2$  (20 mL) was added TBS-Cl (3.2 g, 21.6 mmol). After stirring for 4 h at room temperature, the mixture was poured into sat. aq.  $\text{NH}_4\text{Cl}$  (25 mL), the organic layer was separated, and the aqueous portion was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 10$  mL). The combined organic extracts were washed with saturated NaCl (20 mL) and dried. Removal of the solvent in vacuo followed by column chromatography of the residue afforded pure product.



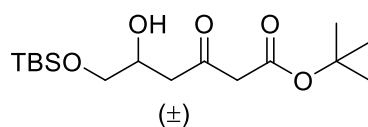
Colorless thick liquid (3.9 g, 83% yield),  $R_f = 0.43$  (5:1 hexanes/ AcOEt).  $^1\text{H NMR}$  (600 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.09–4.05 (m, 2H,  $\text{OCH}_2\text{CH}_3$ ), 3.99–3.98 (m, 1H,  $\text{CHOH}$ ), 3.52–3.51 (m, 2H,  $\text{CH}_2\text{OTBS}$ ), 3.02–3.01 (m, 1H,  $\text{CHOH}$ ), 2.46–2.39 (m, 2H,  $\text{CHOHCH}_2\text{CO}$ ), 1.19–1.17 (m, 3H,  $\text{OCH}_2\text{CH}_3$ ), 0.81 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 0.01 (s, 6H,  $\text{Si}(\text{CH}_3)_2$ ).

**(±)-6-(*tert*-Butyldimethylsilyloxy)-5-hydroxy-3-oxohexanoic acid *tert*-butyl ester**



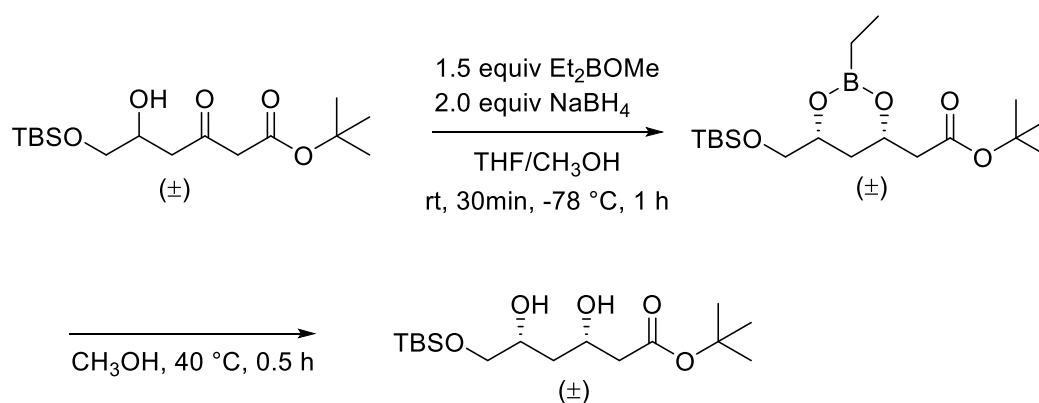
The preparation was following a reported procedure by Carreira (*Angew. Chem., Int. Ed.* **2008**, *47*, 4335.): 4.5 mL (30.5 mmol, 4.0 equiv) of  $\text{iPr}_2\text{NH}$  dissolved in 20 mL of anhydrous THF was cooled to  $-78^\circ\text{C}$  and 19.06 mL (30.5 mmol, 4.0 equiv) of a 1.6 M  $n\text{-BuLi}$  solution in hexane was added via dropping funnel over 45 min. After stirring at  $-78^\circ\text{C}$  for 30 min, 3.54 g (30.5 mmol, 4.0 equiv) of *tert*-butyl acetate was added via dropping funnel over 45 min. The reaction mixture was stirred

for an additional 30 min and then 2 g (7.63 mmol, 1 equiv) of the starting hydroxy ester dissolved in 10 mL THF was added via a dropping funnel over 45 min. The temperature was allowed to rise until  $-55\text{ }^{\circ}\text{C}$  during the addition. The cooling bath was removed and the reaction mixture was allowed to warm to  $-10\text{ }^{\circ}\text{C}$  over 4 h. Then 50 mL of 10% HCl was added over 30 min from a dropping funnel. The temperature was allowed to reach  $10\text{ }^{\circ}\text{C}$  during the addition and the pH reached 2. The reaction mixture was transferred into a separation funnel and the phases were separated. The combined aqueous phases were extracted with EtOAc (30 mL  $\times$  3). The combined organic layers were washed successively with saturated  $\text{NaHCO}_3$  (30 mL) and NaCl (30 mL) before being dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuo to give a viscous pale yellow oil. Flash chromatography with 10:1 hexanes/AcOEt provided the desired product as a colorless oil.



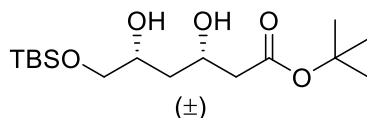
Colorless oil (1.65 g, 64% yield),  $R_f = 0.29$  (5:1 hexanes/ AcOEt).  $^1\text{H NMR}$  (600 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.02–4.01 (m, 1H,  $\text{CHOH}$ ), 3.49–3.47 (m, 2H,  $\text{CH}_2\text{OTBS}$ ), 3.31 (s, 2H,  $\text{COCH}_2\text{CO}$ ), 2.94 (s, 1H, OH), 2.62–2.60 (m, 2H,  $\text{CHOHCH}_2\text{CO}$ ), 1.38–1.29 (s, 9H,  $\text{OC}(\text{CH}_3)_3$ ), 0.78 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ),  $-0.03$  (s, 6H,  $\text{Si}(\text{CH}_3)_2$ ).

**(3*S*\*,5*R*\*)-6-(*tert*-Butyldimethylsilyloxy)-3,5-dihydroxyhexanoic acid *tert*-butyl ester**



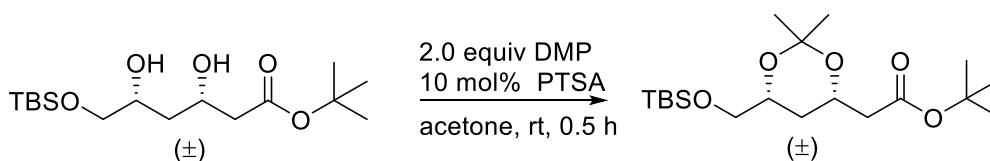
To a solution of the starting  $\beta$ -keto- $\delta$ -hydroxy ester (615 mg, 1.85 mmol) in THF (10 mL) and MeOH (2 mL) was added  $\text{Et}_2\text{BOMe}$  (2.78 mL, 2.78 mmol, 1 mol/L in THF), and the solution was stirred for 30 min at room temperature before being cooled to  $-78\text{ }^{\circ}\text{C}$ .  $\text{NaBH}_4$  (140 mg, 3.7 mmol) was added portionwise over 10 min (moderate gas evolution observed). After the solution was stirred for 1 h at  $-78\text{ }^{\circ}\text{C}$ , 20 mL of sat. aq.  $\text{NH}_4\text{Cl}$  was added to the cold solution. The mixture was

allowed to warm to room temperature and then extracted with EtOAc (50 mL  $\times$  3). The combined organic layers were washed successively with saturated NaCl (20 mL) before being dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo to give a viscous pale yellow oil. MeOH (10 mL) was added, the solution was concentrated via rotary evaporation at ca. 40 °C. This was repeated for 4 times. The mixture was concentrated and purified by column chromatography.

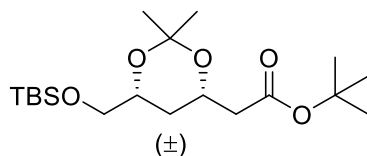


Colorless oil (587 mg, 95% yield),  $R_f$  = 0.29 (5:1 hexanes/ AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$ : 4.19–4.18 (s, 1H, CHOH), 3.85 (s, 2H, CH<sub>2</sub>OTBS), 3.52–3.45 (m, 2H, two CHOH), 3.26–3.25 (m, 1H, OH), 2.43–2.33 (m, 2H, CH<sub>2</sub>COOC(CH<sub>3</sub>)<sub>3</sub>), 1.63–1.47 (m, 2H, OHCHCH<sub>2</sub>CHOH), 1.41 (s, 9H, OC(CH<sub>3</sub>)<sub>3</sub>), 0.85 (s, 9H, SiC(CH<sub>3</sub>)<sub>3</sub>), 0.022 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>).

**[(4*S*\*,6*R*\*)-6-(*tert*-Butyldimethylsilyloxymethyl-2,2-dimethyl-1,3-dioxan-4-yl)]acetic acid *tert*-butyl ester**

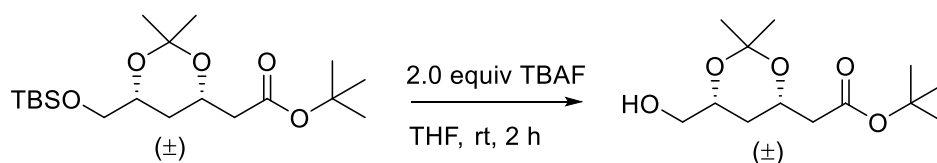


To a solution of the dihydroxy ester (861 mg, 2.57 mmol) in acetone (6 mL) were added 2,2-dimethoxypropane (536 mg, 5.1 mmol) and *p*-toluenesulfonic acid monohydrate (44 mg, 0.26 mmol, 0.1 equiv). The resulting mixture was stirred at room temperature until TLC analysis indicated consumption of the starting material (0.5 h). Et<sub>3</sub>N (3 mL) was added, and the mixture was concentrated and purified by column chromatography.

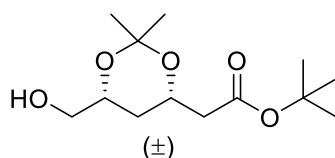


Colorless oil (904 mg, 94% yield),  $R_f$  = 0.67 (5:1 hexanes/ AcOEt). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$ : 4.21–4.20 (m, 1H, one of CH<sub>2</sub>OTBS), 3.89–3.87 (m, 1H, one of CH<sub>2</sub>OTBS), 3.63–3.39 (m, 2H, OCHCH<sub>2</sub>CHO), 2.41–2.23 (m, 2H, CH<sub>2</sub>COOC(CH<sub>3</sub>)<sub>3</sub>), 1.61–1.59 (m, 1H, one of OCHCH<sub>2</sub>CHO), 1.40–1.39 (m, 9H, OC(CH<sub>3</sub>)<sub>3</sub>), 1.31 (s, 6H, C(CH<sub>3</sub>)<sub>3</sub>), 1.15–1.09 (m, 1H, one of OCHCH<sub>2</sub>CHO), 0.83 (s, 9H, C(CH<sub>3</sub>)<sub>3</sub>), –0.01 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>).

**[(4*S*\*,6*R*\*)-2,2-Dimethyl-6-hydroxymethyl-1,3-dioxan-4-yl]acetic acid *tert*-butyl ester**

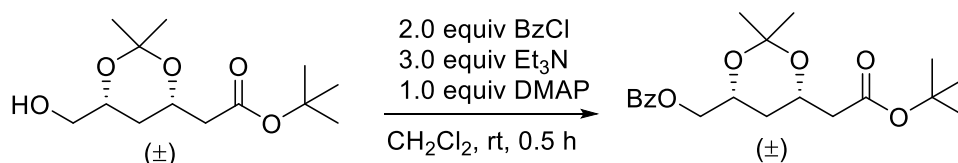


To a stirred solution of the TBS ether (581 mg, 1.55 mmol) in THF (4 mL) was added 1 M tetrabutylammonium fluoride in THF (3.1 mL, 3.1 mmol, 2 equiv), and the reaction mixture was stirred at room temperature for 2 h. The reaction was quenched with aqueous  $\text{NH}_4\text{Cl}$ . The layers were separated, and the aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine and dried over  $\text{Na}_2\text{SO}_4$ . After evaporation of the solvent under reduced pressure, the residue was purified by column chromatography.

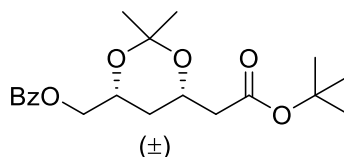


Colorless oil (346 mg, 86% yield),  $R_f = 0.42$  (1:1 hexanes/ AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.24–3.89 (m, 2H,  $\text{OHCH}_2$ ), 3.52–3.39 (m, 2H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$ ), 2.62 (s, 1H, OH), 2.39–2.20 (m, 2H,  $\text{CH}_2\text{COOC}(\text{CH}_3)_3$ ), 1.44–1.41 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ), 1.39 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.36 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 1.30 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.26–1.17 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ).

**[(4*S*\*,6*R*\*)-6-Benzoyloxymethyl-2,2-dimethyl-1,3-dioxan-4-yl]acetic acid *tert*-butyl ester**



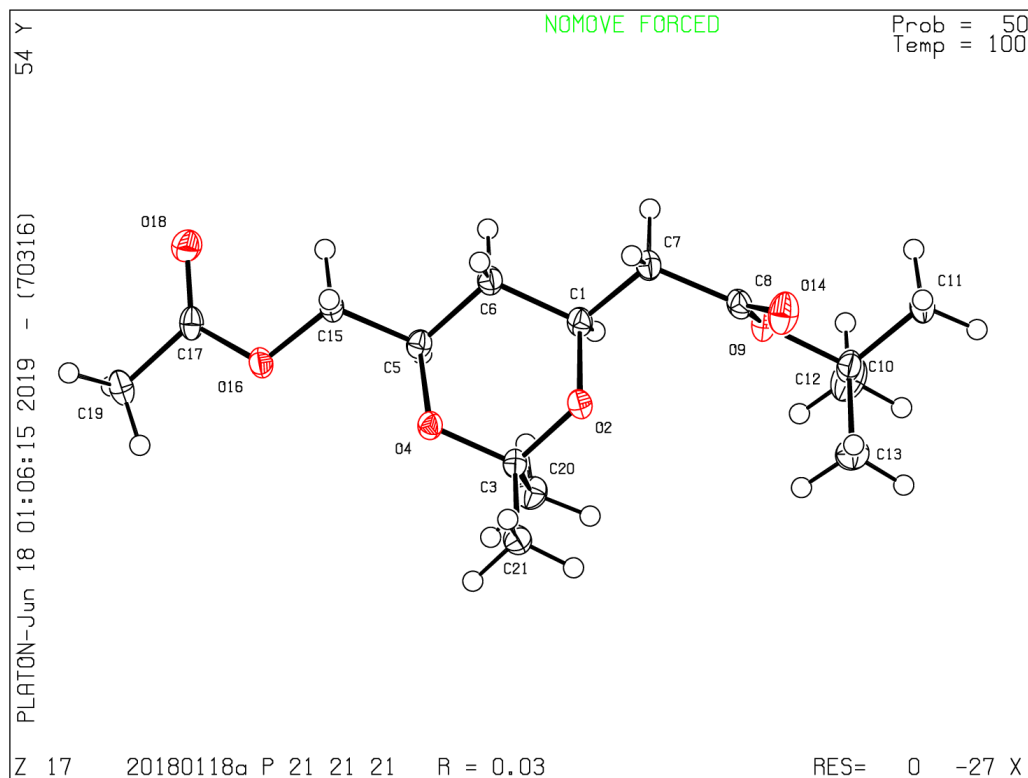
To a solution of the starting material (82 mg, 0.31 mmol) in  $\text{CH}_2\text{Cl}_2$  (4 mL) were added  $\text{Et}_3\text{N}$  (130  $\mu\text{L}$ , 0.95 mmol), DMAP (38 mg, 0.31 mmol) and benzoyl chloride (71  $\mu\text{L}$ , 0.62 mmol). After stirring for 0.5 h at room temperature, the mixture was purified by column chromatography directly.



Colorless oil (103 mg, 92% yield),  $R_f = 0.37$  (5:1 hexanes/ AcOEt).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 8.04–8.02 (m, 2H, ArH), 7.56–7.52 (m 1H, ArH), 7.44–7.41 (m, 2H, ArH), 4.32–4.23 (m, 4H,  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{CHOC}(\text{CH}_3)_2$  and  $\text{BzOCH}_2$ ), 2.48–2.31 (m, 2H,  $\text{CH}_2\text{COOC}(\text{CH}_3)_3$ ), 1.68–1.64 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ) 1.48 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.43 (s, 9H,  $\text{C}(\text{CH}_3)_3$ ), 1.40 (s, 3H,  $\text{C}(\text{CH}_3)_2$ ), 1.38–1.32 (m, 1H, one of  $\text{OCHCH}_2\text{CHO}$ ).

### 3. Crystallographic data of compound 17

Crystals of compound 17 were grown in hexanes at 4 °C by slow evaporation of the solvents. The ellipsoid contour percent probability level was 50%.



Bond precision:	C-C = 0.0029 Å		Wavelength=1.54184
Cell:	a=5.6812(2)	b=12.9135(4)	c=22.1615(6)
	alpha=90	beta=90	gamma=90
Temperature:	100 K		
	Calculated	Reported	
Volume	1625.86(9)	1625.86(9)	
Space group	P 21 21 21	P 21 21 21	
Hall group	P 2ac 2ab	P 2ac 2ab	
Moiety formula	C15 H26 O6	C15 H26 O6	
Sum formula	C15 H26 O6	C15 H26 O6	
Mr	302.36	302.36	
Dx,g cm-3	1.235	1.235	
Z	4	4	
Mu (mm-1)	0.785	0.785	
F000	656.0	656.0	



F000'	658.20	
h,k,lmax	7,16,27	6,15,27
Nref	3255[ 1908]	3203
Tmin,Tmax	0.954,0.962	0.453,1.000
Tmin'	0.855	

Correction method= # Reported T Limits: Tmin=0.453 Tmax=1.000 AbsCorr = MULTI-SCAN

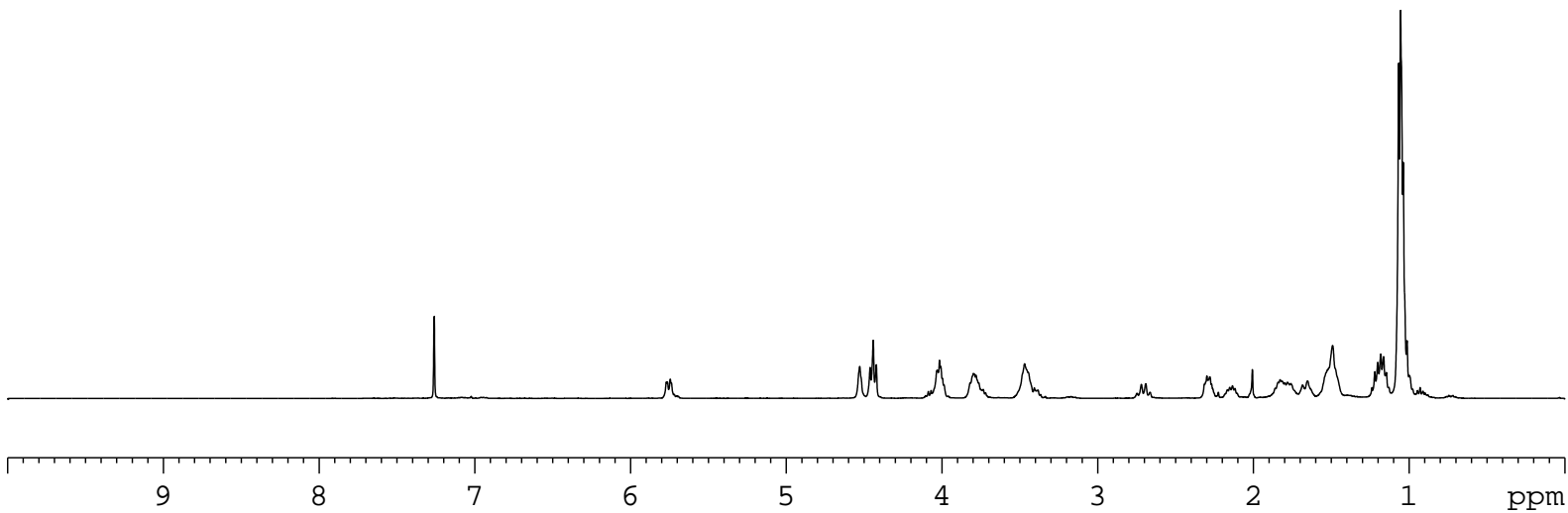
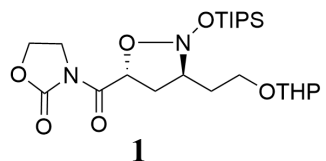
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R(reflections)= 0.0339( 3043)                      wR2(reflections)= 0.0891( 3203)

S = 1.086                      Npar= 196

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3.03  
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1.38  
0.91  
2.46  
1.31  
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NAME JMH161215  
EXPNO 1  
PROCNO 1  
Date\_ 20161217  
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TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 40.3  
DW 60.800 usec  
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TE 291.6 K  
D1 1.00000000 sec  
TD0 1

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SF01 400.1724712 MHz  
SI 32768  
SF 400.1700025 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

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— 153.14

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98.89

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69.75

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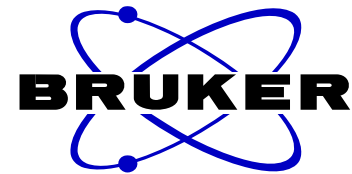
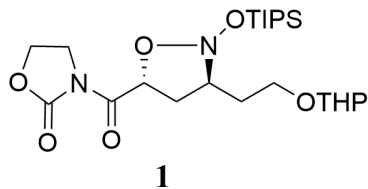
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18.05

18.02

12.20



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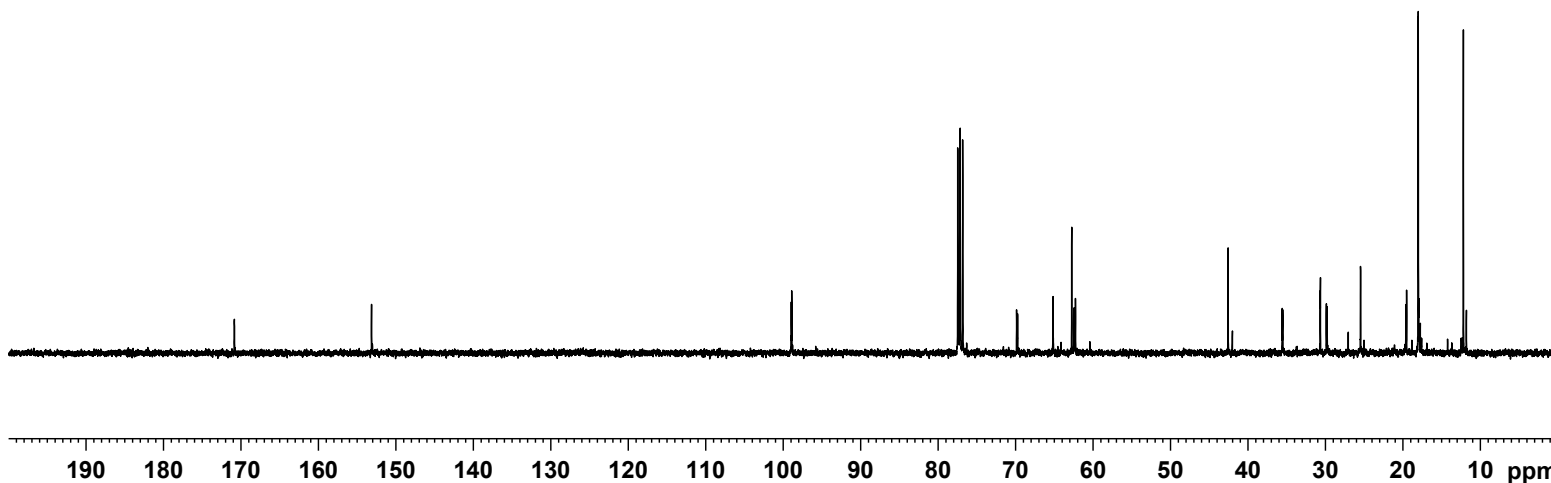
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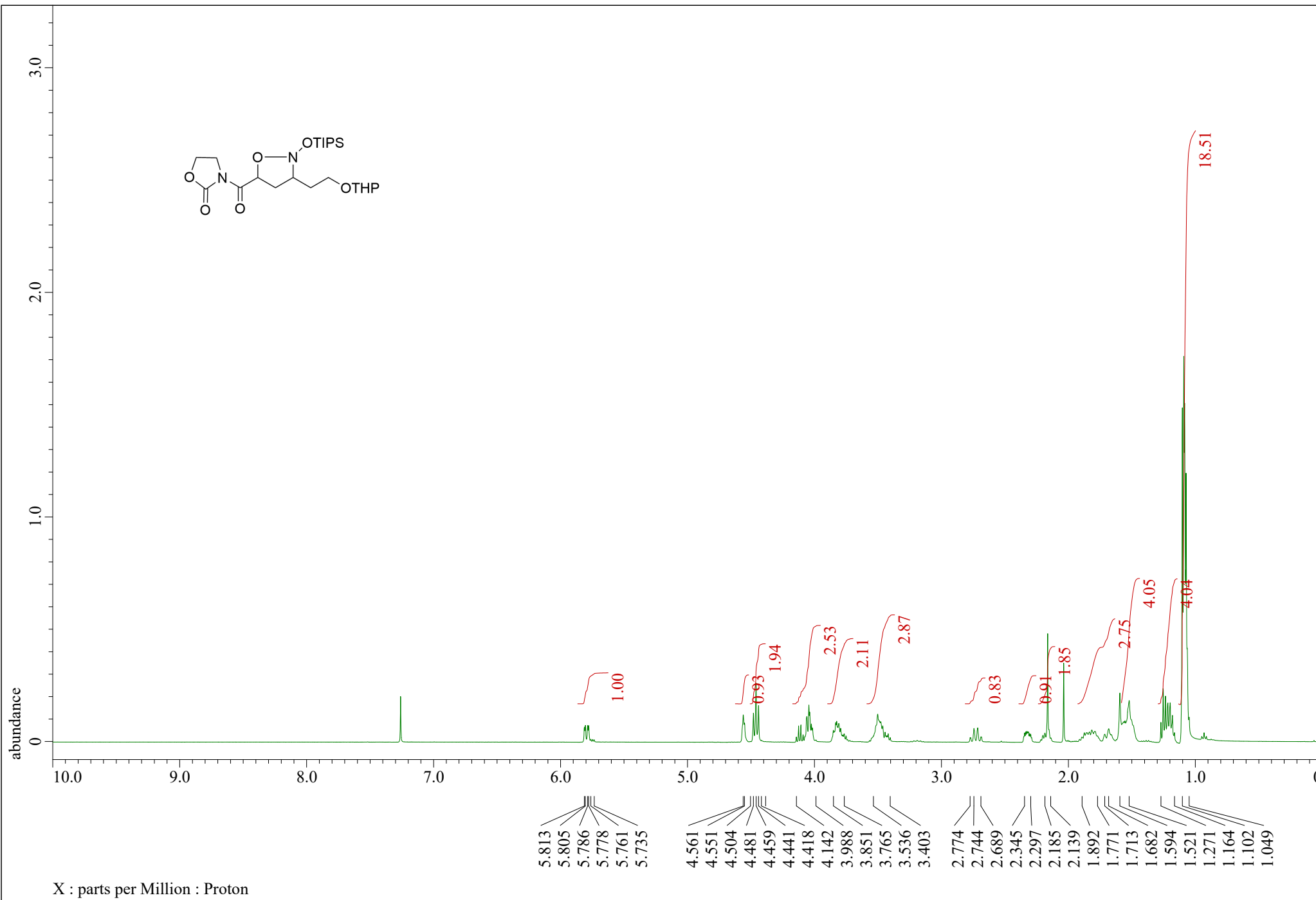
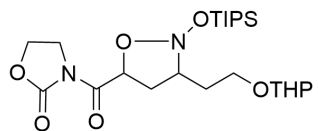
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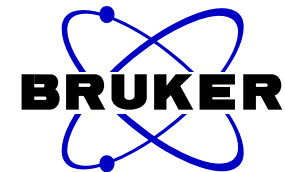
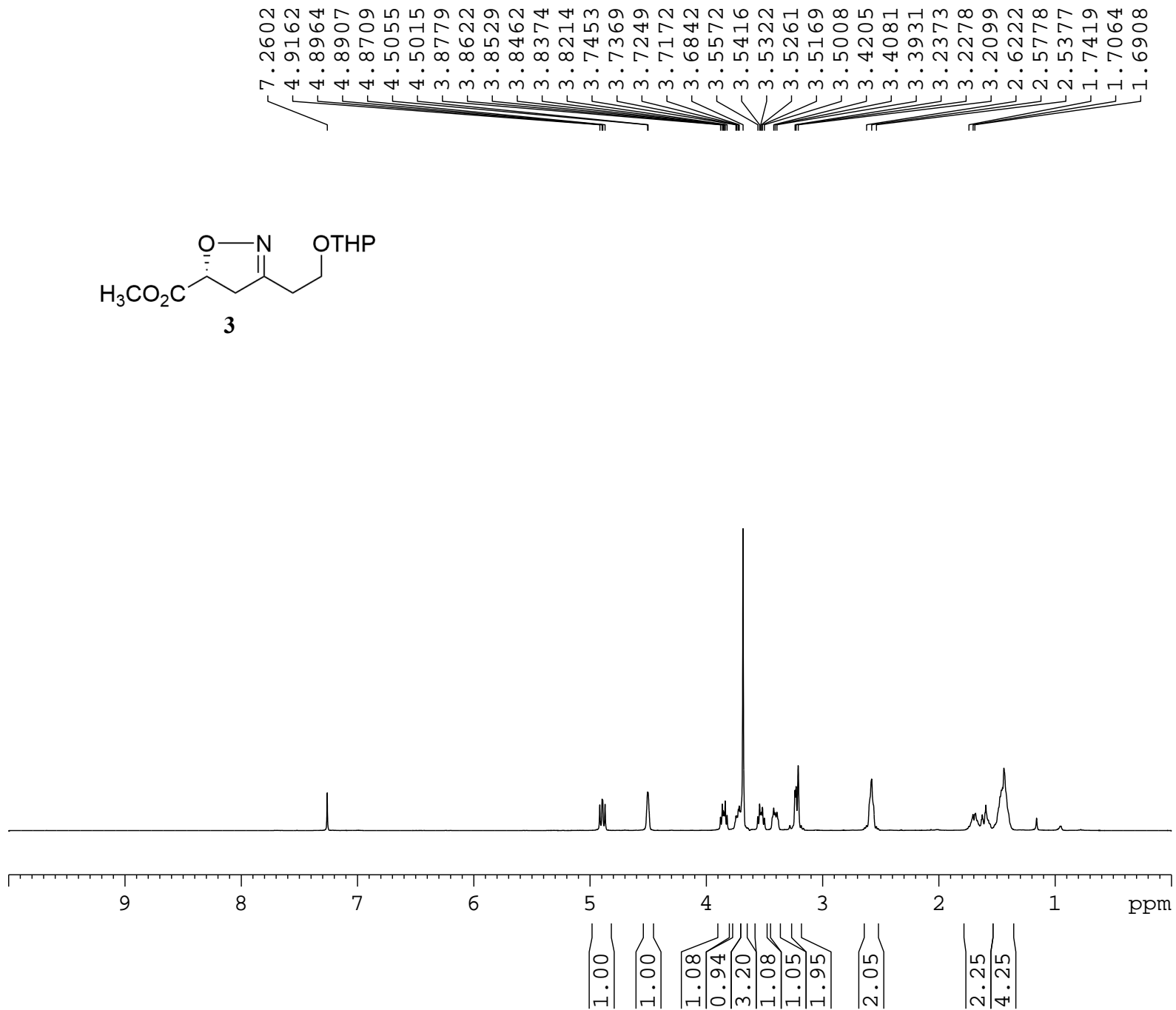
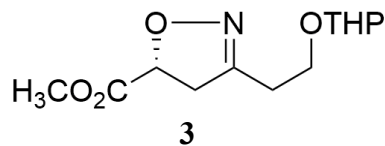
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PL12      14.26 dB
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PL2W      13.18669796 W
PL12W     0.39276794 W
PL13W     0.37509048 W
SFO2      400.1716007 MHz
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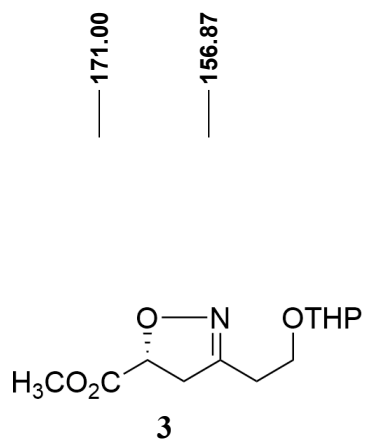
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SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        36
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DE        6.50 usec
TE        301.3 K
D1        1.00000000 sec
TD0       1

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===== CHANNEL f1 =====
NUC1      1H
P1        13.80 usec
PL1       -1.00 dB
PL1W     13.18669796 W
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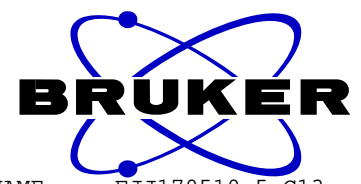
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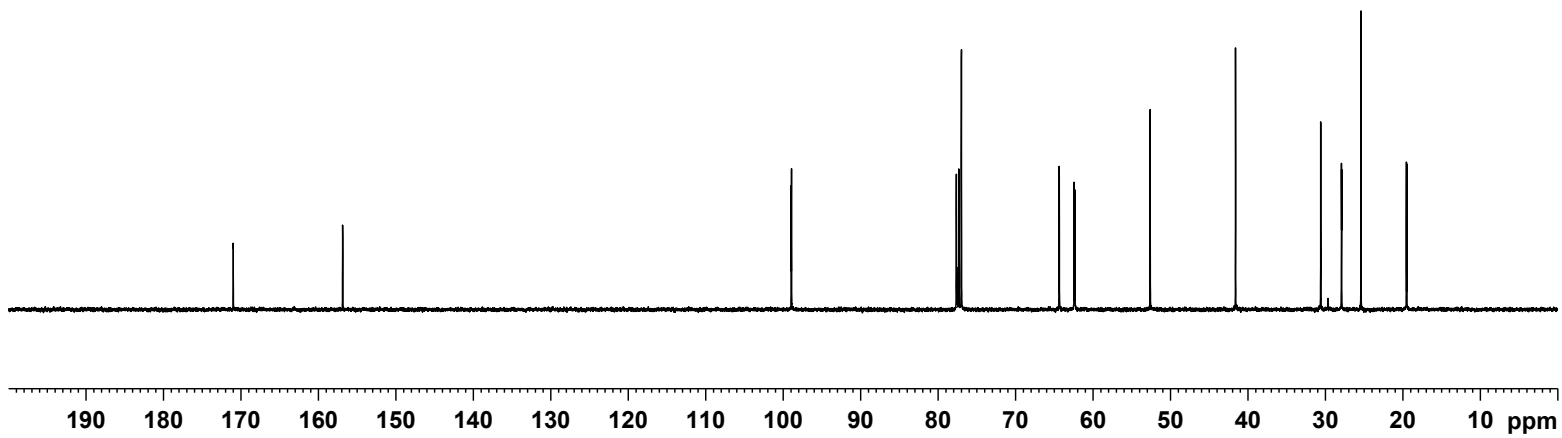
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NS        137
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AQ        1.3631988 sec
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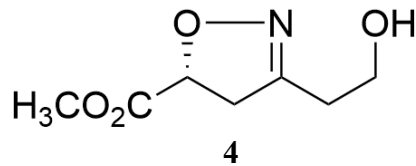
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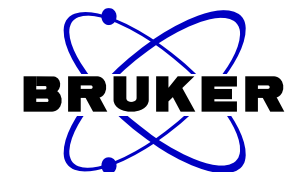
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PL12      14.26 dB
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PL12W     0.39276794 W
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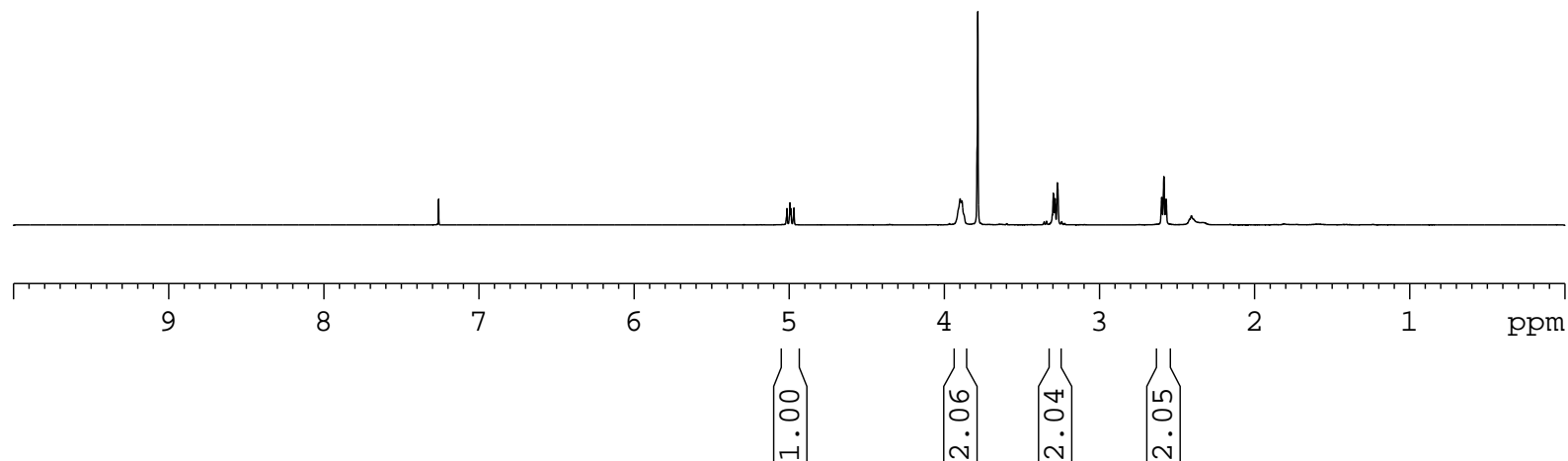


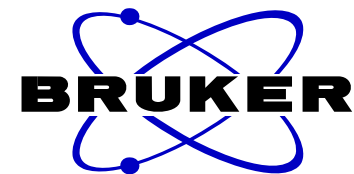
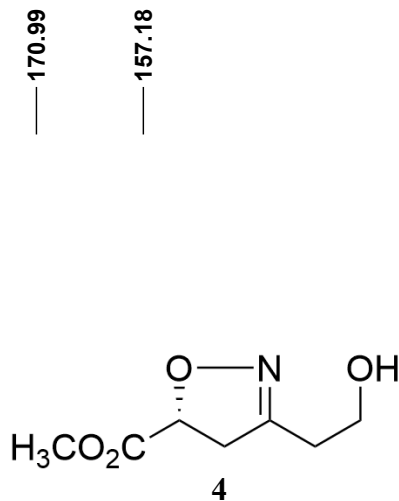
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DE         6.50 usec
TE         294.6 K
D1         1.0000000 sec
TD0        1
  
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PL1        -1.00 dB
PL1W       13.18669796 W
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PROCNO    1
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TD        65536
SOLVENT   CDC13
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FIDRES    0.366798 Hz
AQ        1.3631988 sec
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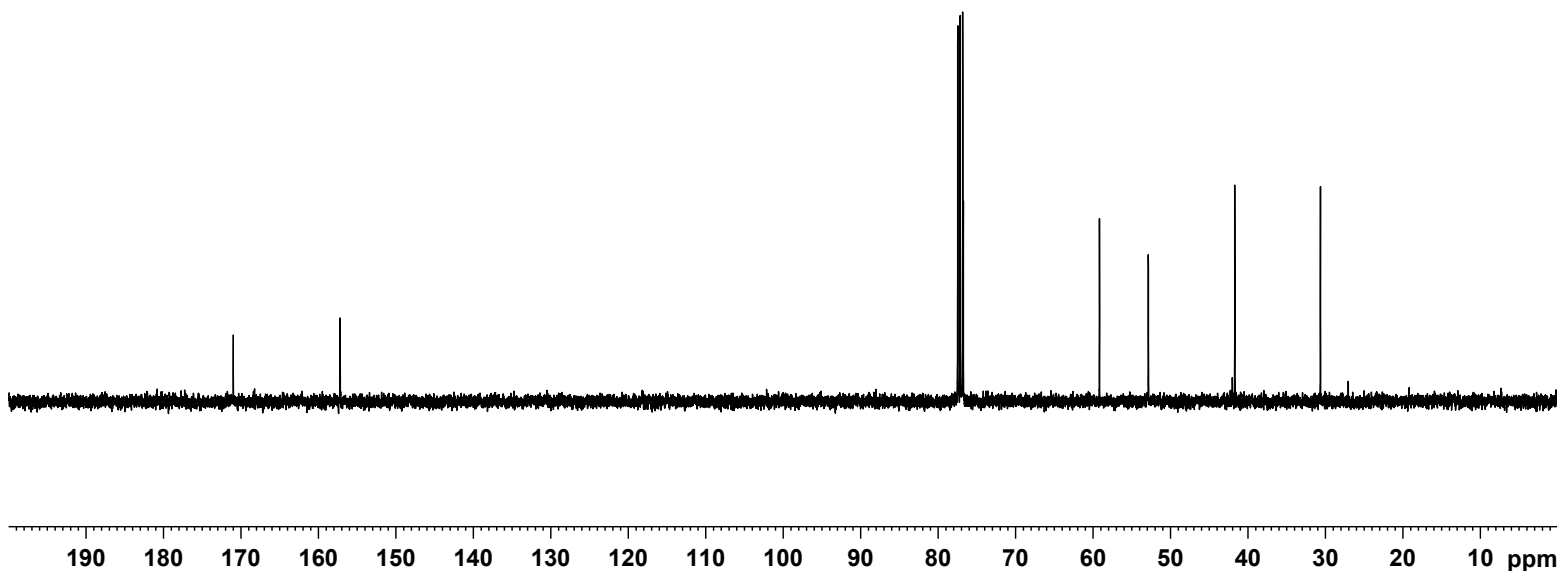
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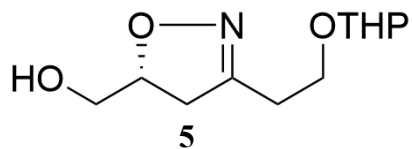
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PL2       -1.00 dB
PL12      14.26 dB
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PL2W      13.18669796 W
PL12W     0.39276794 W
PL13W     0.37509048 W
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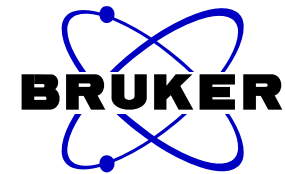
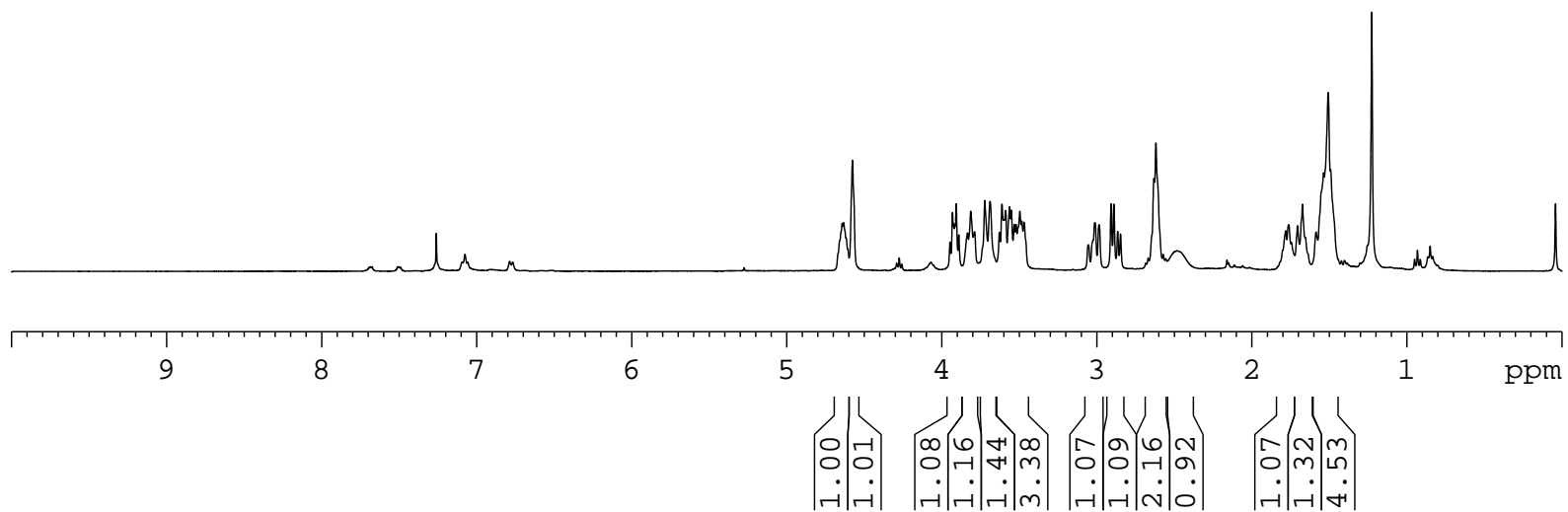
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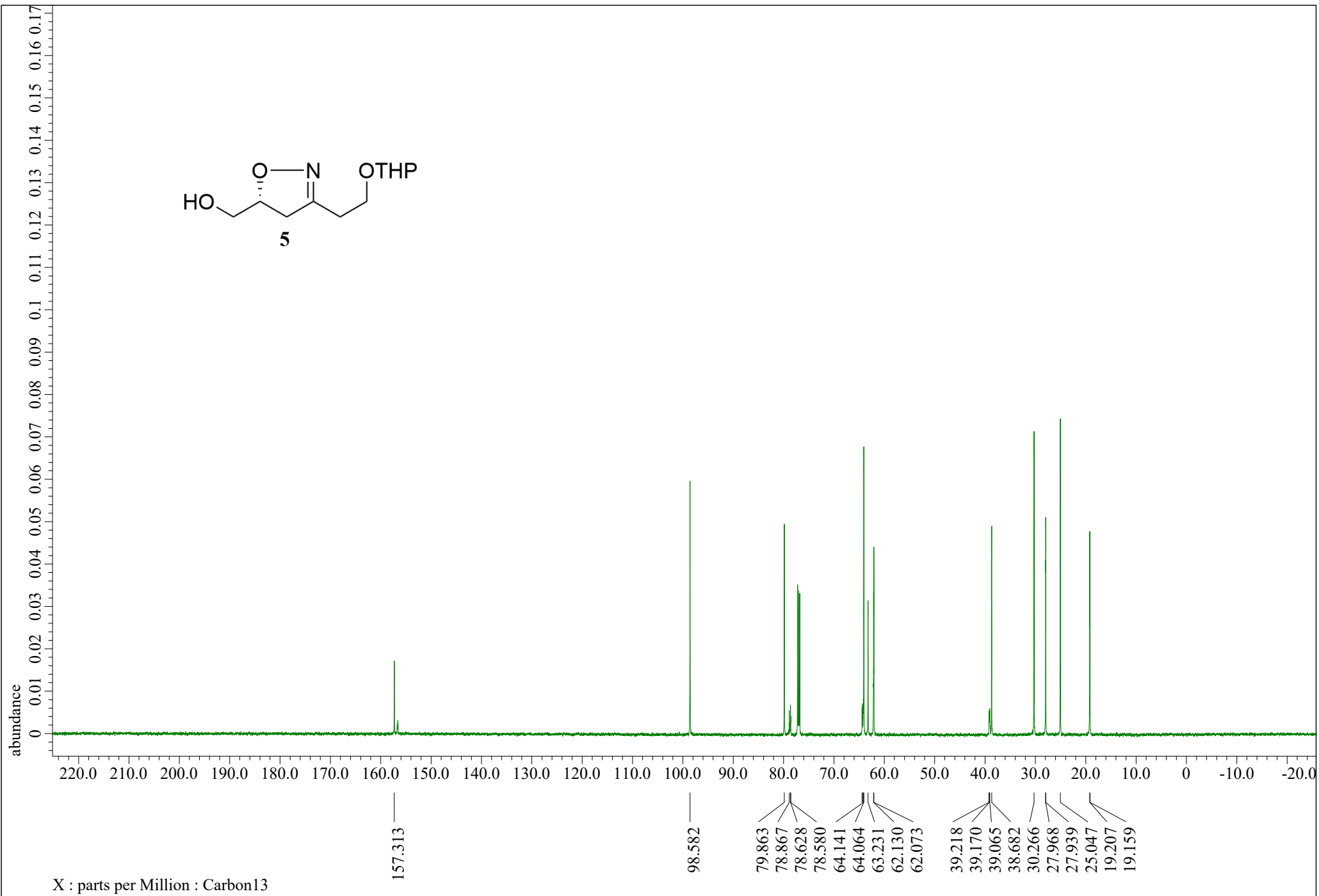
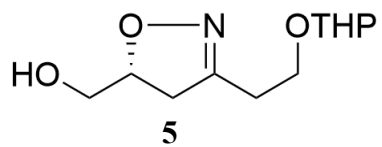


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2.6185  
2.4832

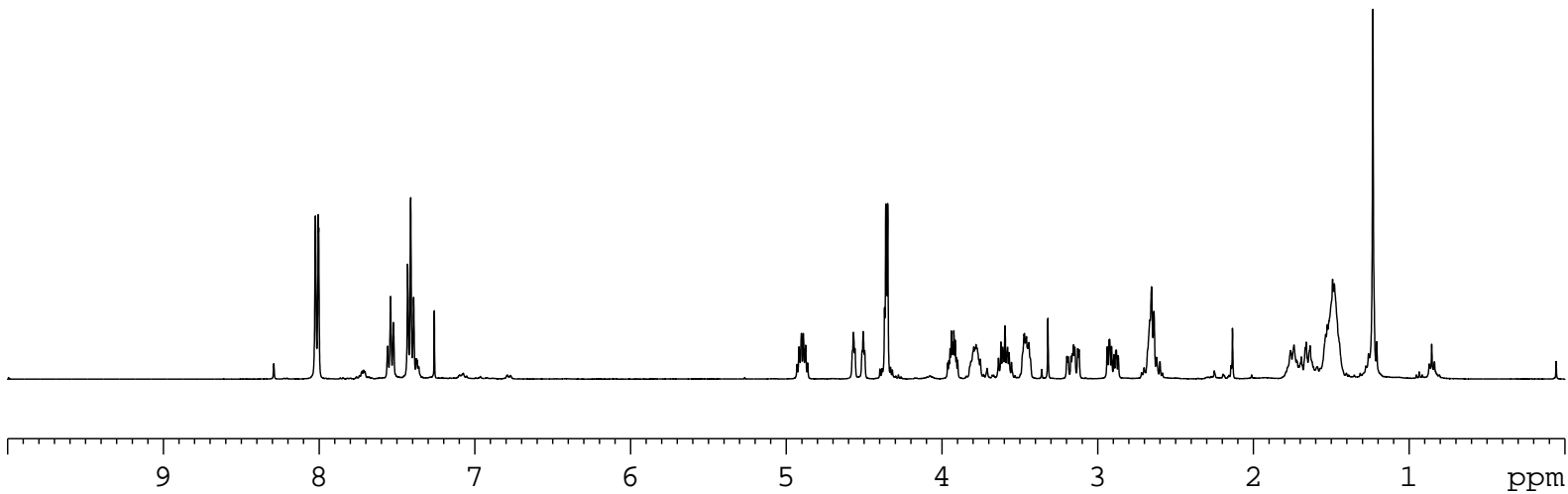
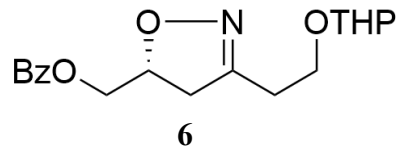


NAME FJJ170328  
EXPNO 1  
PROCNO 1  
Date\_ 20170329  
Time 14.49  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 64  
DW 60.800 usec  
DE 6.50 usec  
TE 299.2 K  
D1 1.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 13.80 usec  
PL1 -1.00 dB  
PL1W 13.18669796 W  
SF01 400.1724712 MHz  
SI 32768  
SF 400.1700028 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



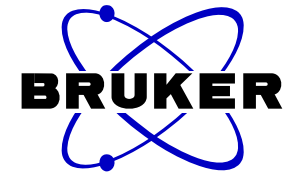
8.0234  
8.0053  
8.0026  
7.5598  
7.5412  
7.5228  
7.4313  
7.4118  
7.3928  
4.9027  
4.8906  
4.8754  
4.5687  
4.5061  
4.3688  
4.3608  
4.3486  
3.9395  
3.9315  
3.9237  
3.9152  
3.7814  
3.6210  
3.5955  
3.5786  
3.4738  
3.4705  
3.4638  
3.4575  
3.4508  
3.4422  
3.1553  
3.1467  
2.9390  
2.9230  
2.9108  
2.6638  
2.6544  
2.6388  
1.7392  
1.6609  
1.6360  
1.5359  
1.5253  
1.4915  
1.4805



2.00  
1.04  
2.11

1.02  
1.03  
2.07  
1.05  
1.11  
1.18  
1.21  
1.04  
1.06  
2.13

1.07  
1.12  
4.33

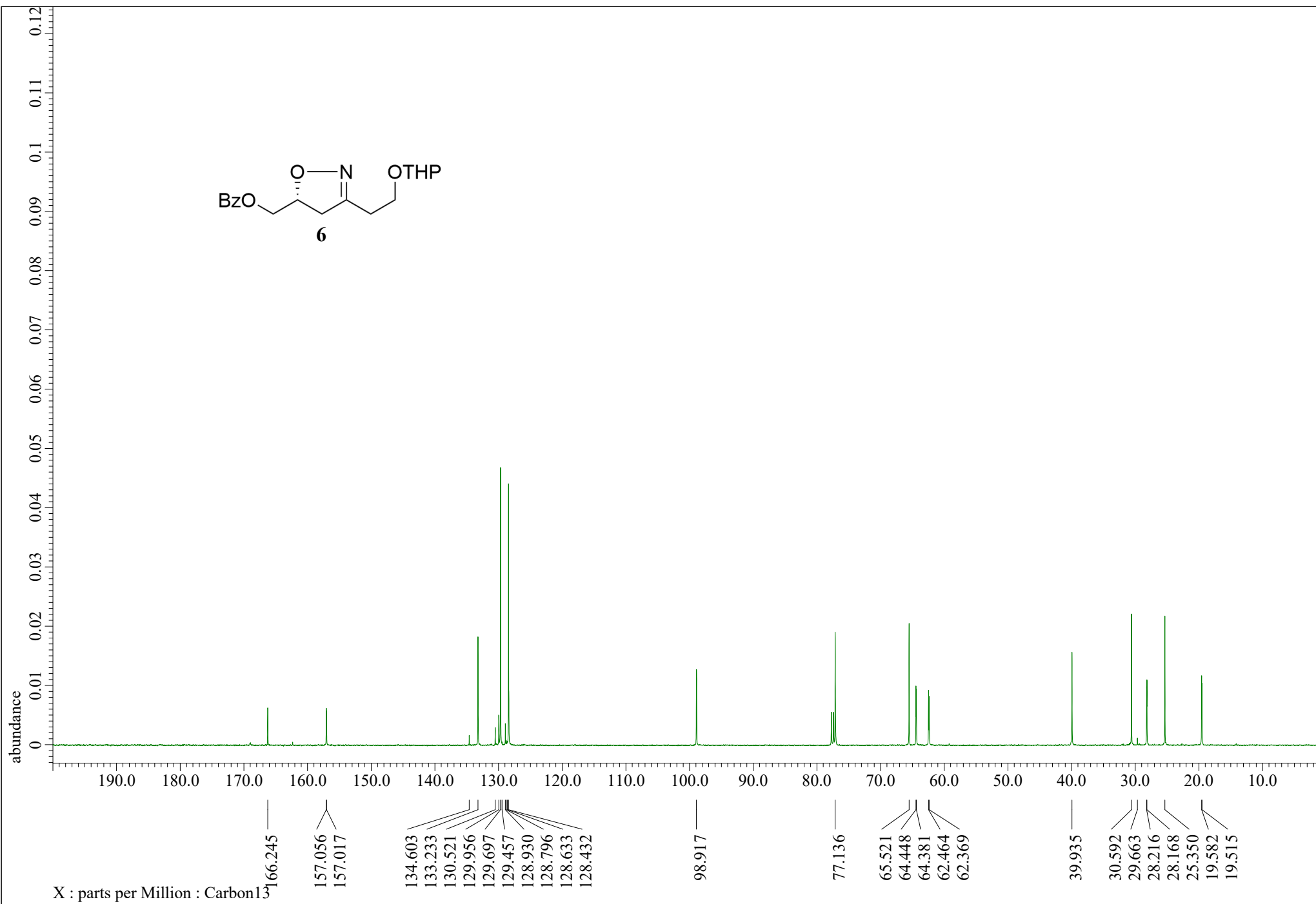
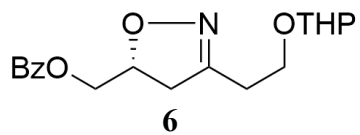


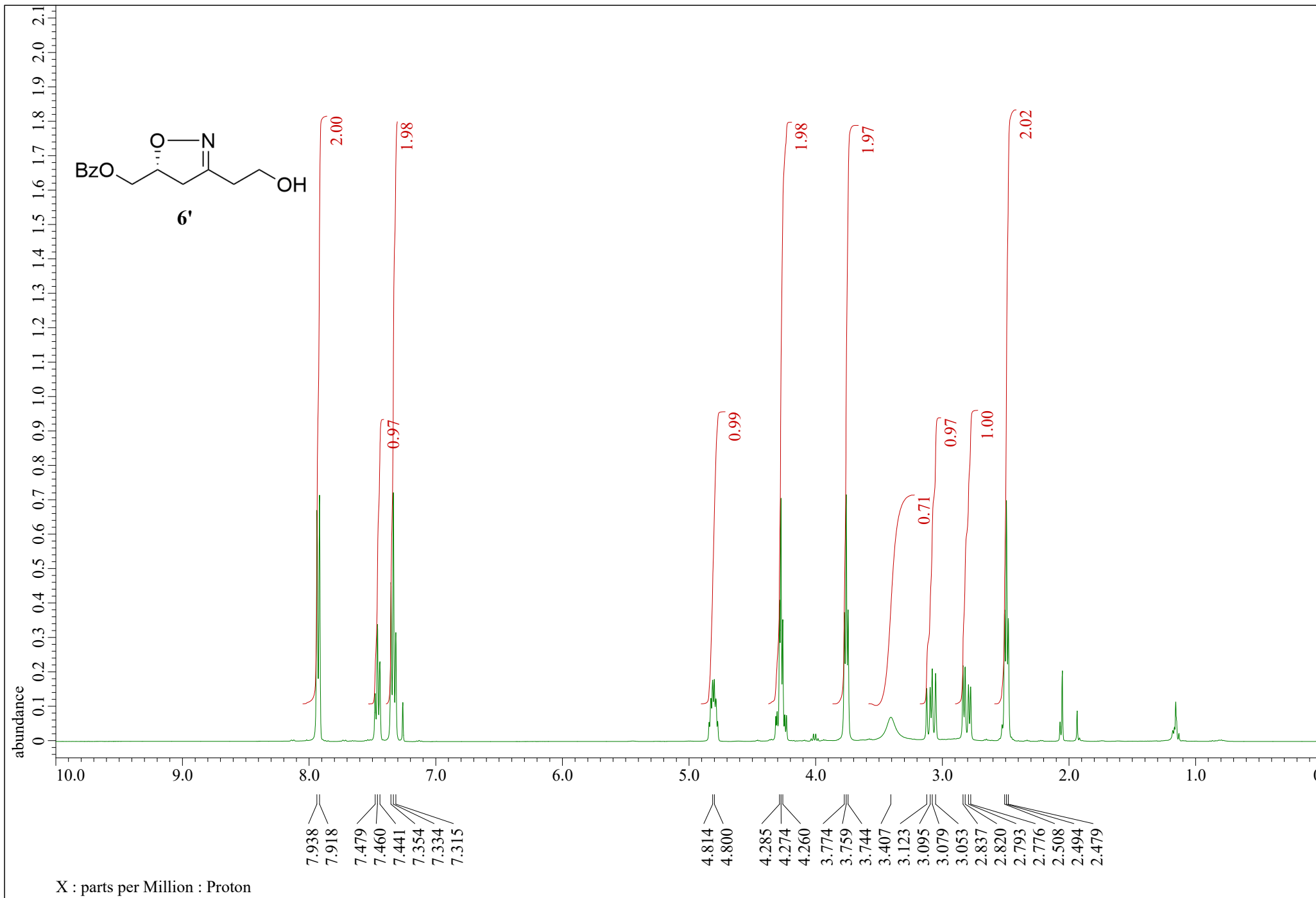
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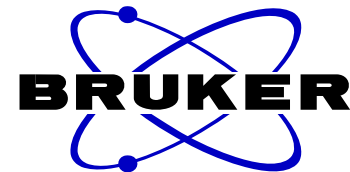
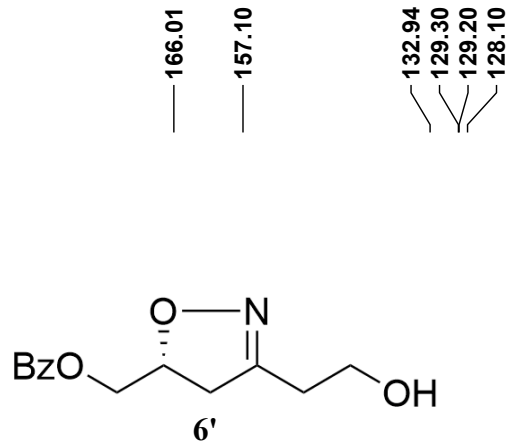
NAME          FJJ170330
EXPNO         1
PROCNO       1
Date_        20170331
Time         22.02
INSTRUM     spect
PROBHD      5 mm PABBO BB-
PULPROG     zg30
TD          65536
SOLVENT     CDCl3
NS           16
DS           2
SWH         8223.685 Hz
FIDRES      0.125483 Hz
AQ          3.9846387 sec
RG           50.8
DW          60.800 usec
DE           6.50 usec
TE          297.3 K
D1          1.0000000 sec
TD0         1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1           13.80 usec
PL1          -1.00 dB
PL1W         13.18669796 W
SF01         400.1724712 MHz
SI           32768
SF           400.1700028 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



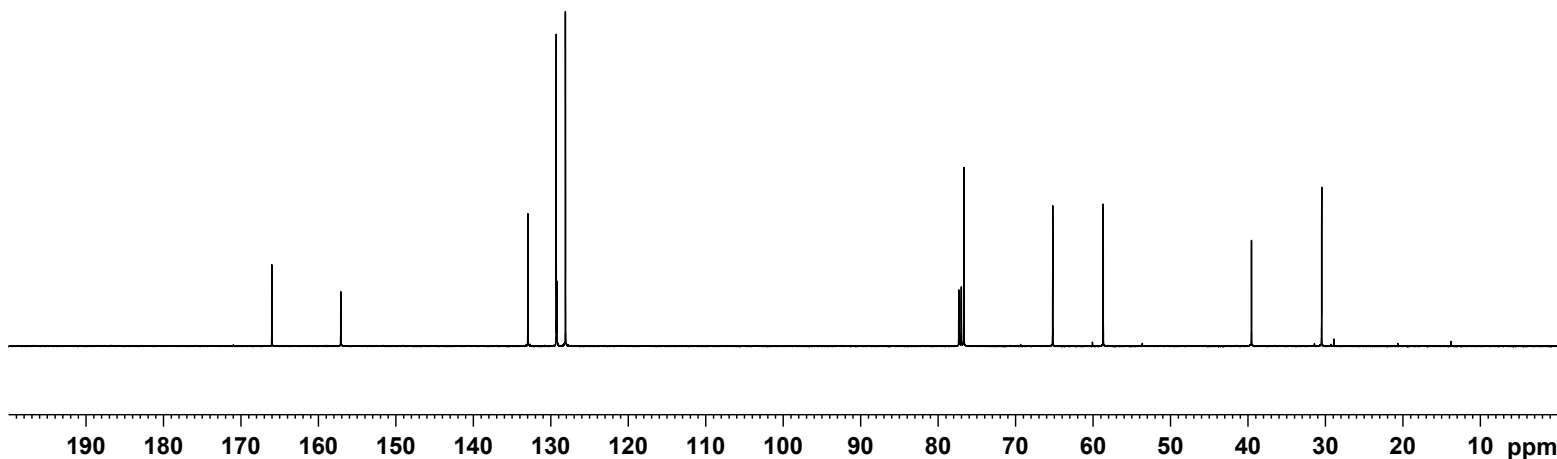




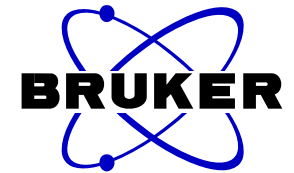
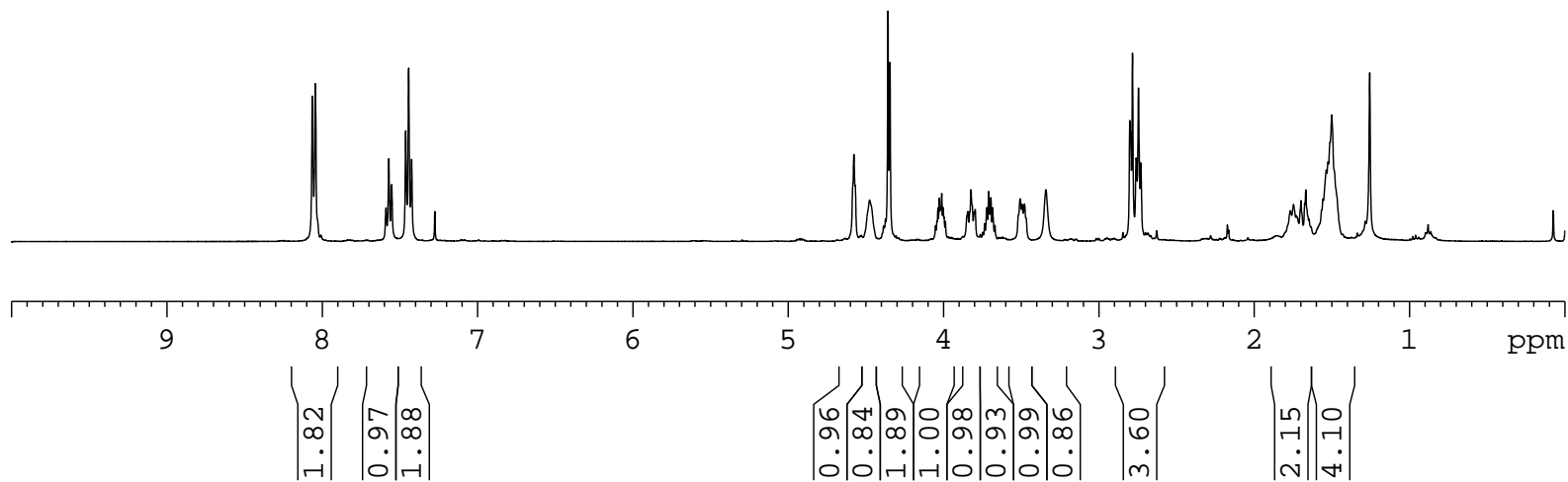
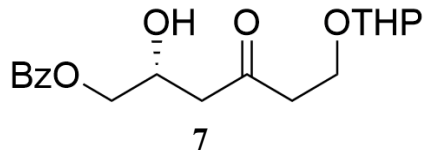
NAME FJJ170724-C13  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20170728  
 Time 11.57  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 400  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 203  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 299.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 =====  
 NUC1 13C  
 P1 8.50 usec  
 PL1 -2.00 dB  
 PL1W 57.32743073 W  
 SFO1 100.6328888 MHz

==== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -1.00 dB  
 PL12 13.26 dB  
 PL13 14.46 dB  
 PL2W 13.18669796 W  
 PL12W 0.49446553 W  
 PL13W 0.37509048 W  
 SFO2 400.1716007 MHz  
 SI 32768  
 SF 100.6228581 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



8.0633  
8.0448  
7.5897  
7.5713  
7.5528  
7.4629  
7.4435  
7.4246  
4.5772  
4.5687  
4.4760  
4.3575  
4.3450  
4.0362  
4.0268  
4.0172  
4.0116  
4.0027  
3.8438  
3.8231  
3.7959  
3.7211  
3.7094  
3.6949  
3.6839  
3.5069  
3.4936  
3.4788  
3.3408  
2.8009  
2.7959  
2.7832  
2.7597  
2.7446  
2.7297  
1.7676  
1.7467  
1.6983  
1.6744  
1.6675  
1.5572  
1.5359  
1.5243  
1.5010  
1.2561  
0.0750

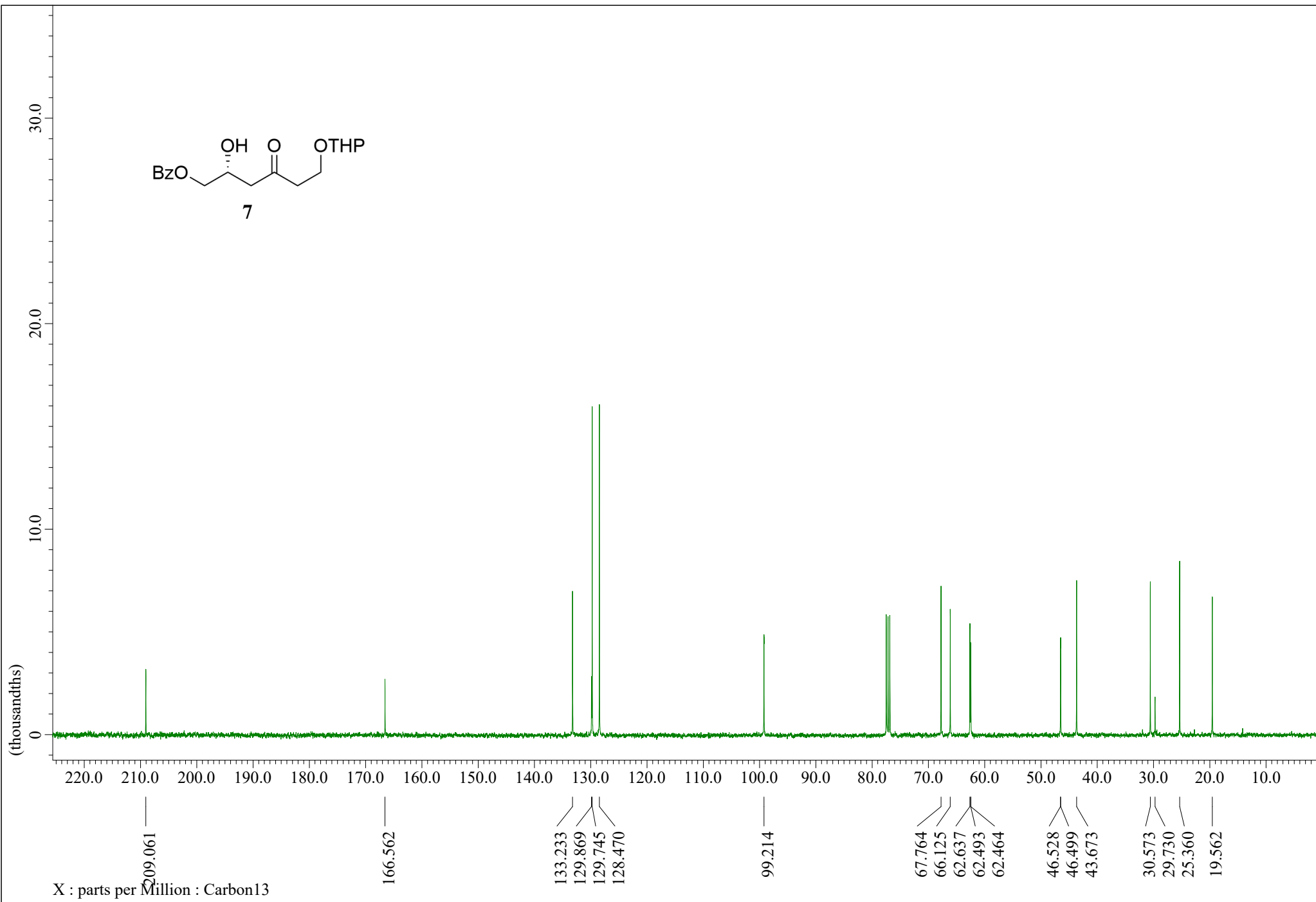
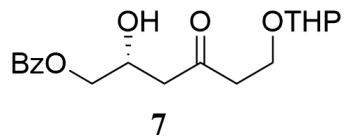


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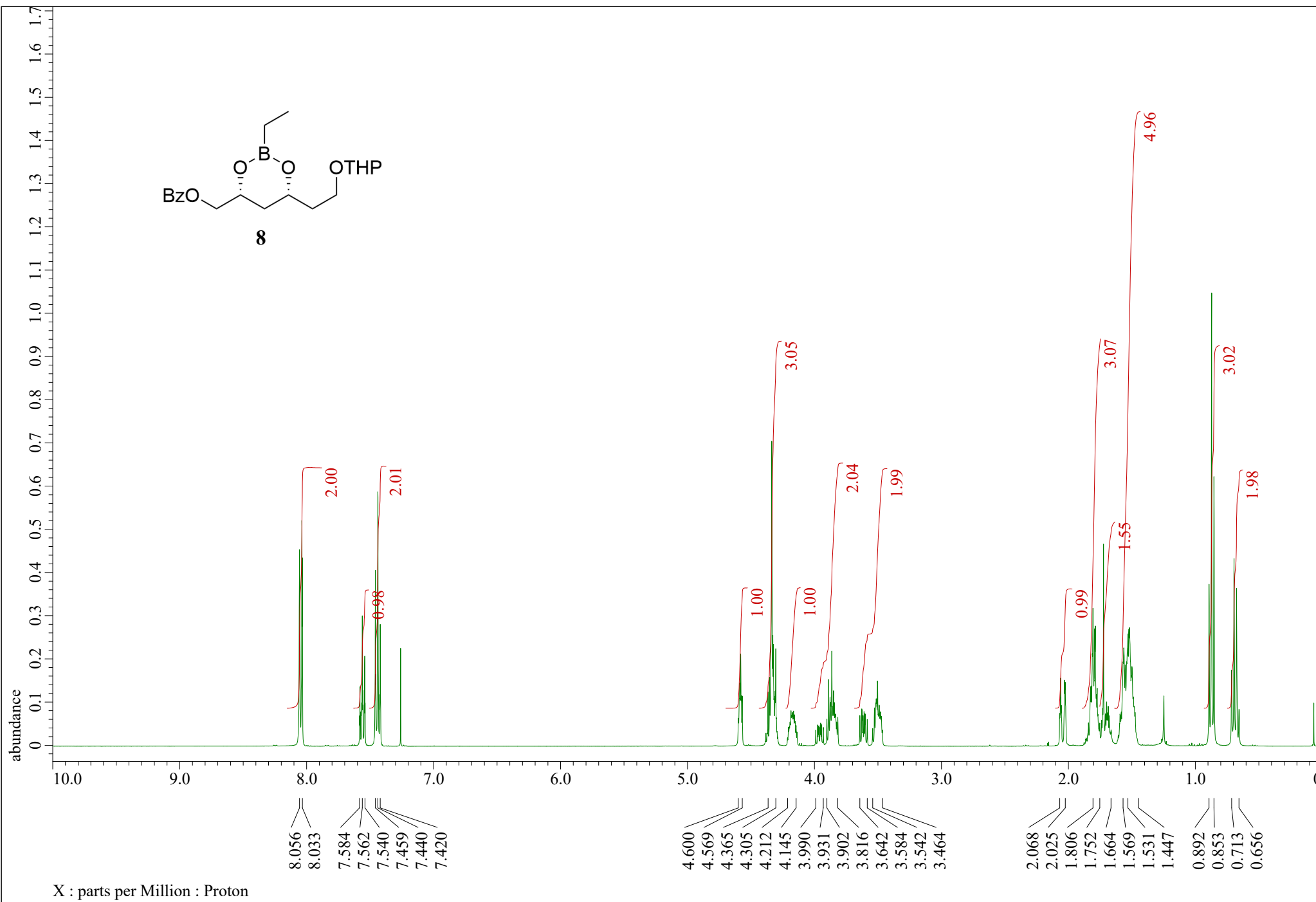
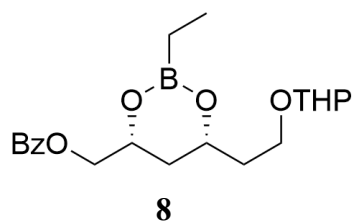
NAME          FJJ170413-XD
EXPNO         1
PROCNO        1
Date_         20170413
Time          21.24
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            80.6
DW            60.800 usec
DE            6.50 usec
TE            297.6 K
D1            1.00000000 sec
TDO           1
  
```

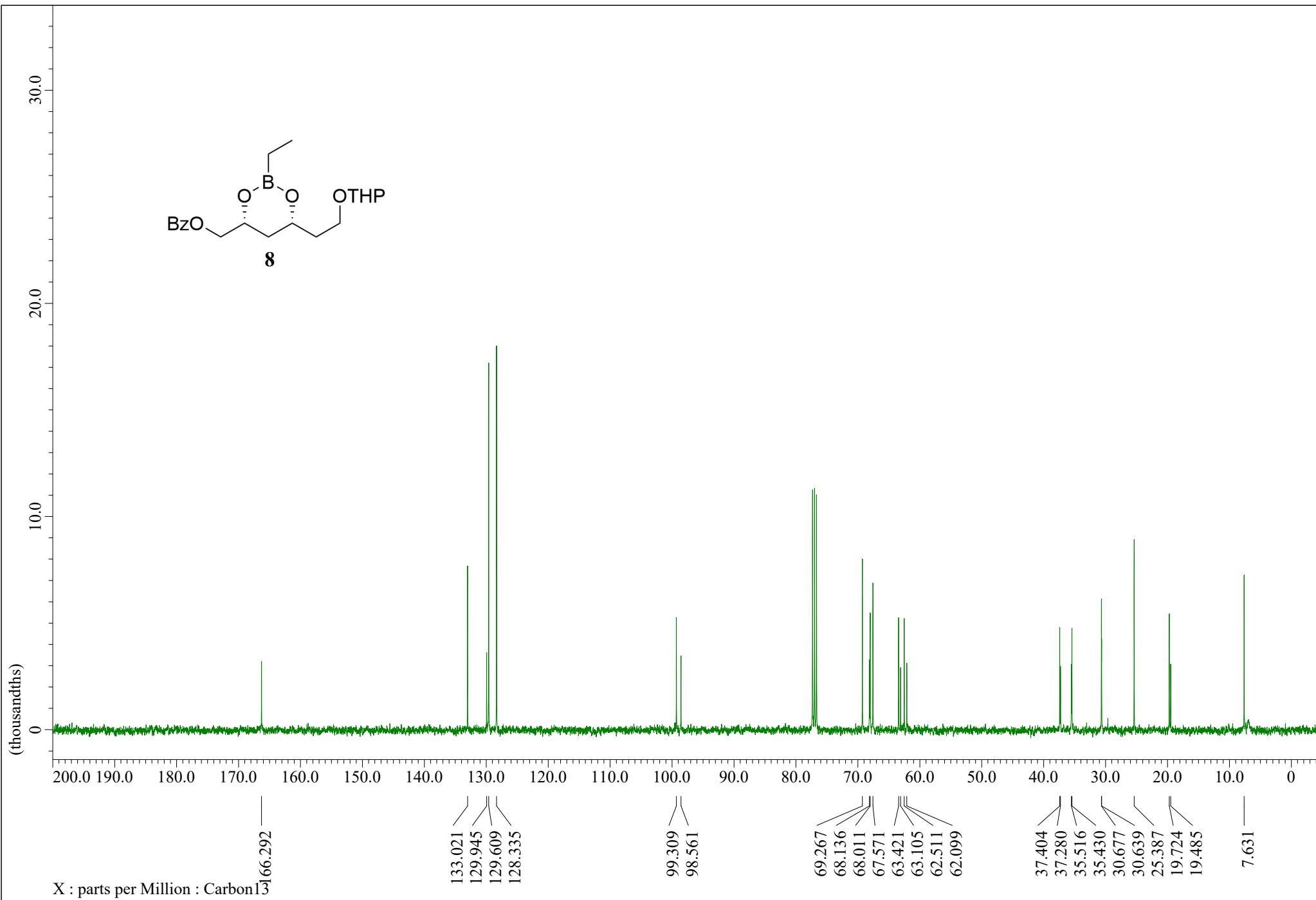
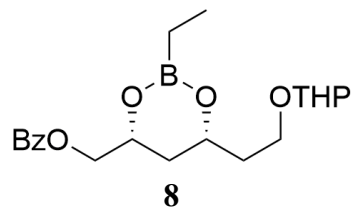
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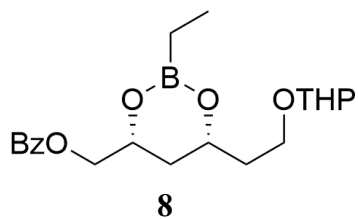
===== CHANNEL f1 =====
NUC1           1H
P1             13.80 usec
PL1            -1.00 dB
PL1W          13.18669796 W
SF01          400.1724712 MHz
SI             32768
SF            400.1699966 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
  
```





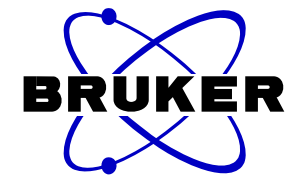






—31.4583

—-0.0020

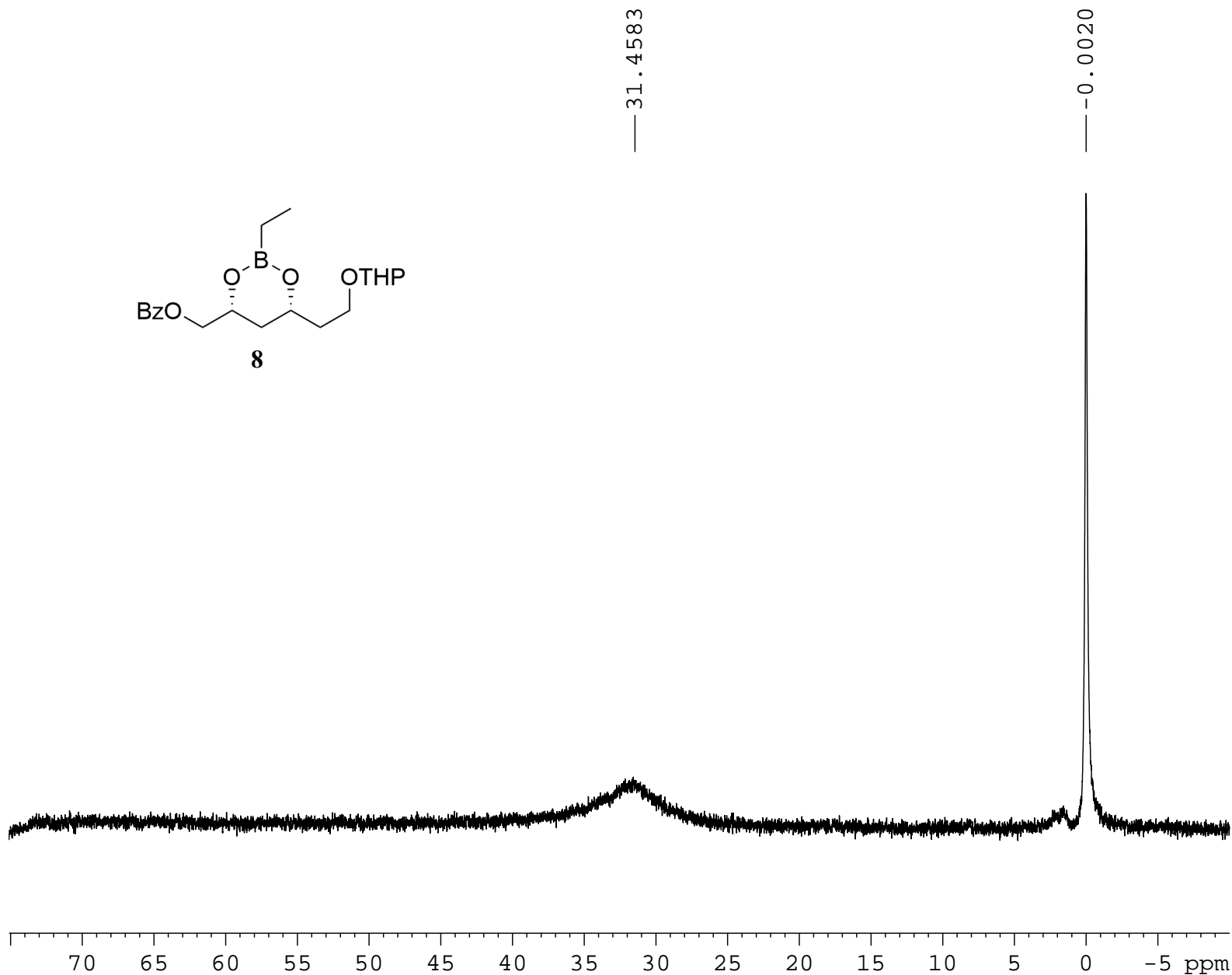


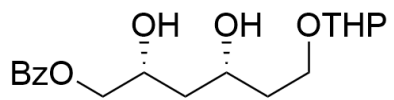
```

NAME      FJJ171207-2-B
EXPNO     1
PROCNO    1
Date_     20171207
Time      13.25
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg
TD         65536
SOLVENT   CDCl3
NS         2992
DS         0
SWH        19230.770 Hz
FIDRES     0.293438 Hz
AQ         1.7039860 sec
RG         203
DW         26.000 usec
DE         6.50 usec
TE         296.7 K
D1         1.00000000 sec
TD0        1
  
```

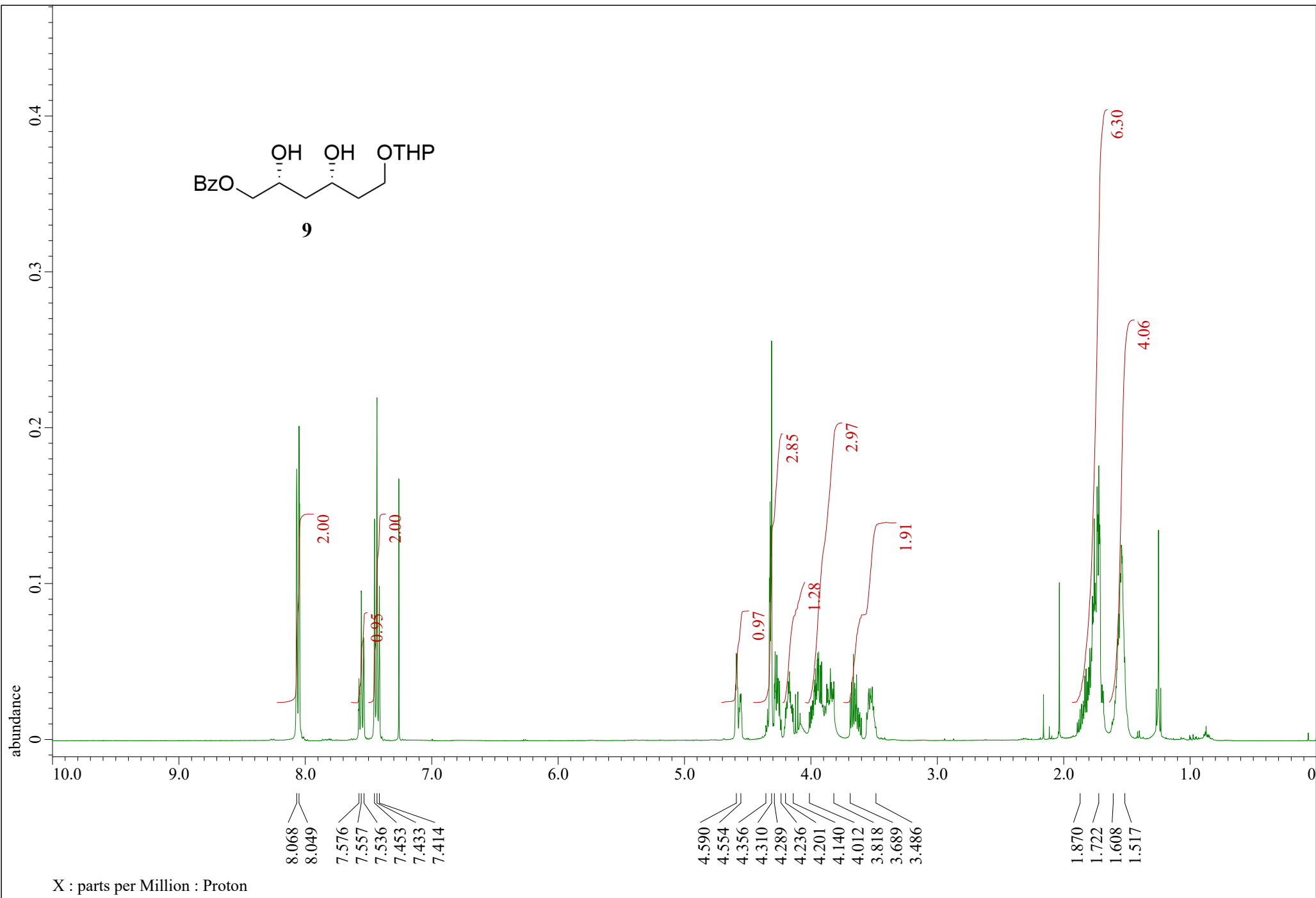
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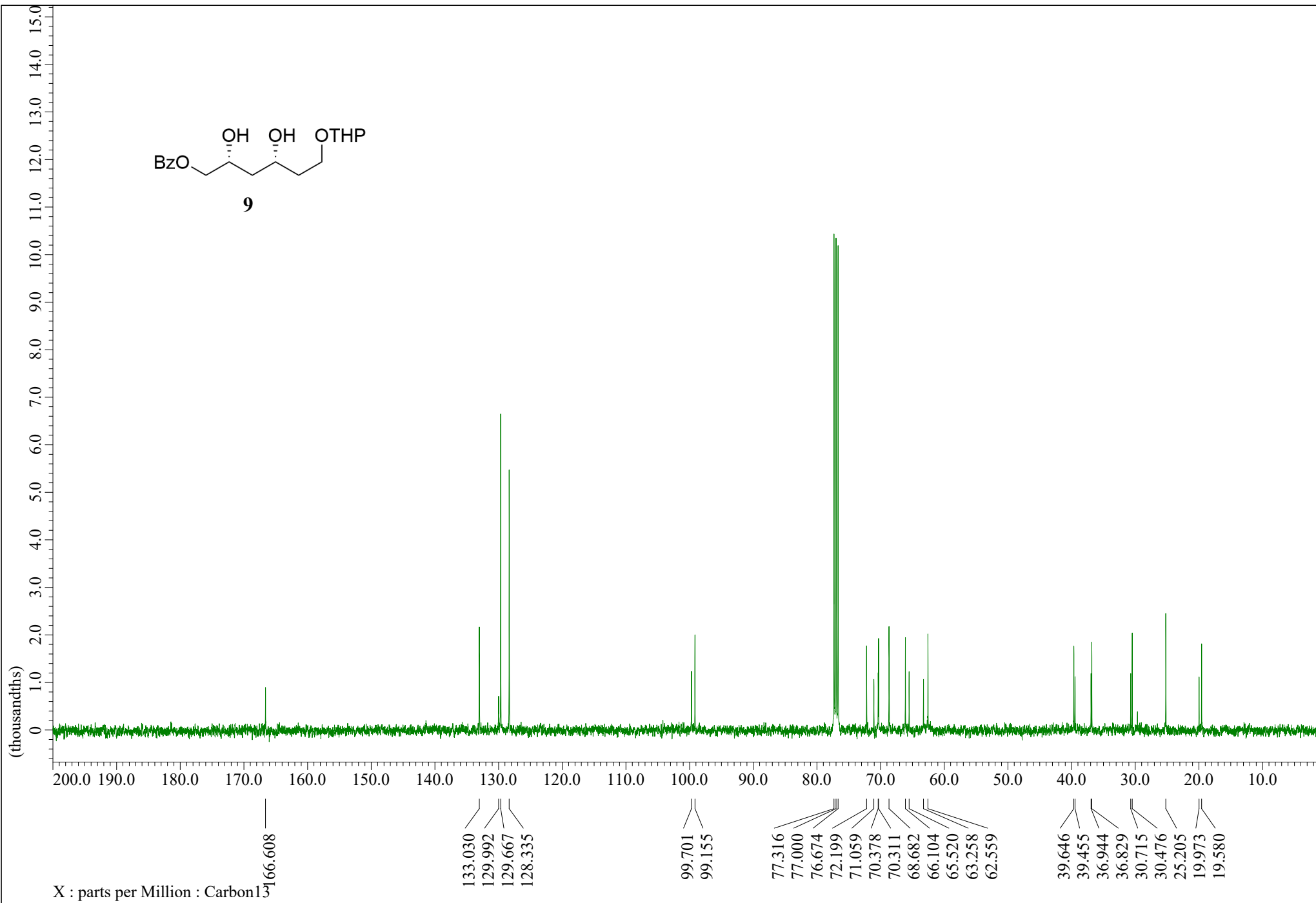
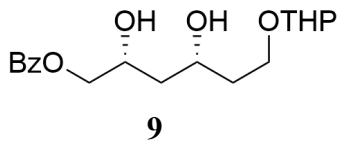
===== CHANNEL f1 =====
NUC1       11B
P1         8.00 usec
PL1        -2.00 dB
SFO1       128.3904390 MHz
SI         32768
SF         128.3904126 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

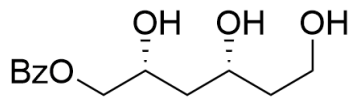




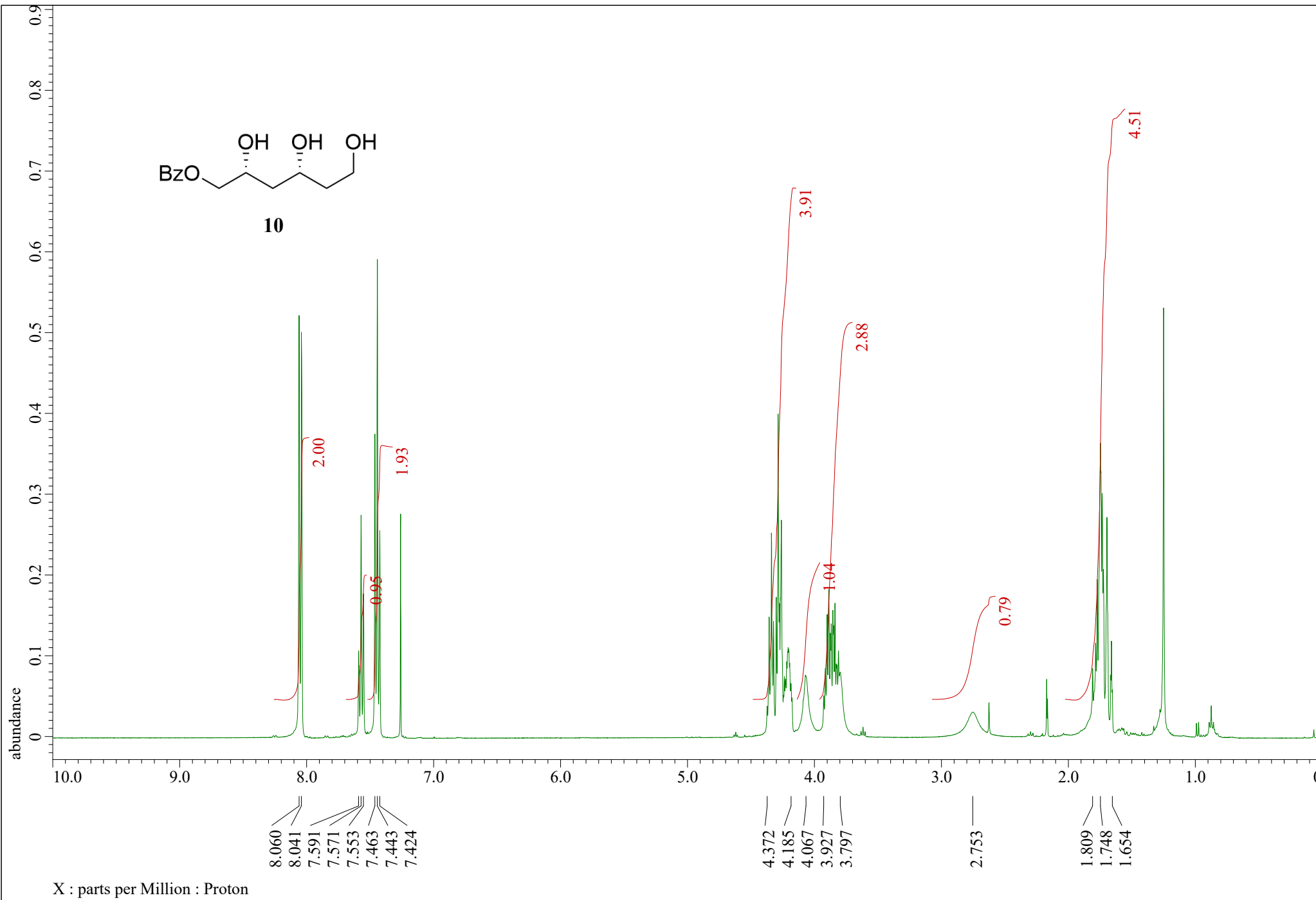
9

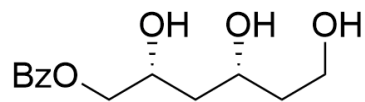




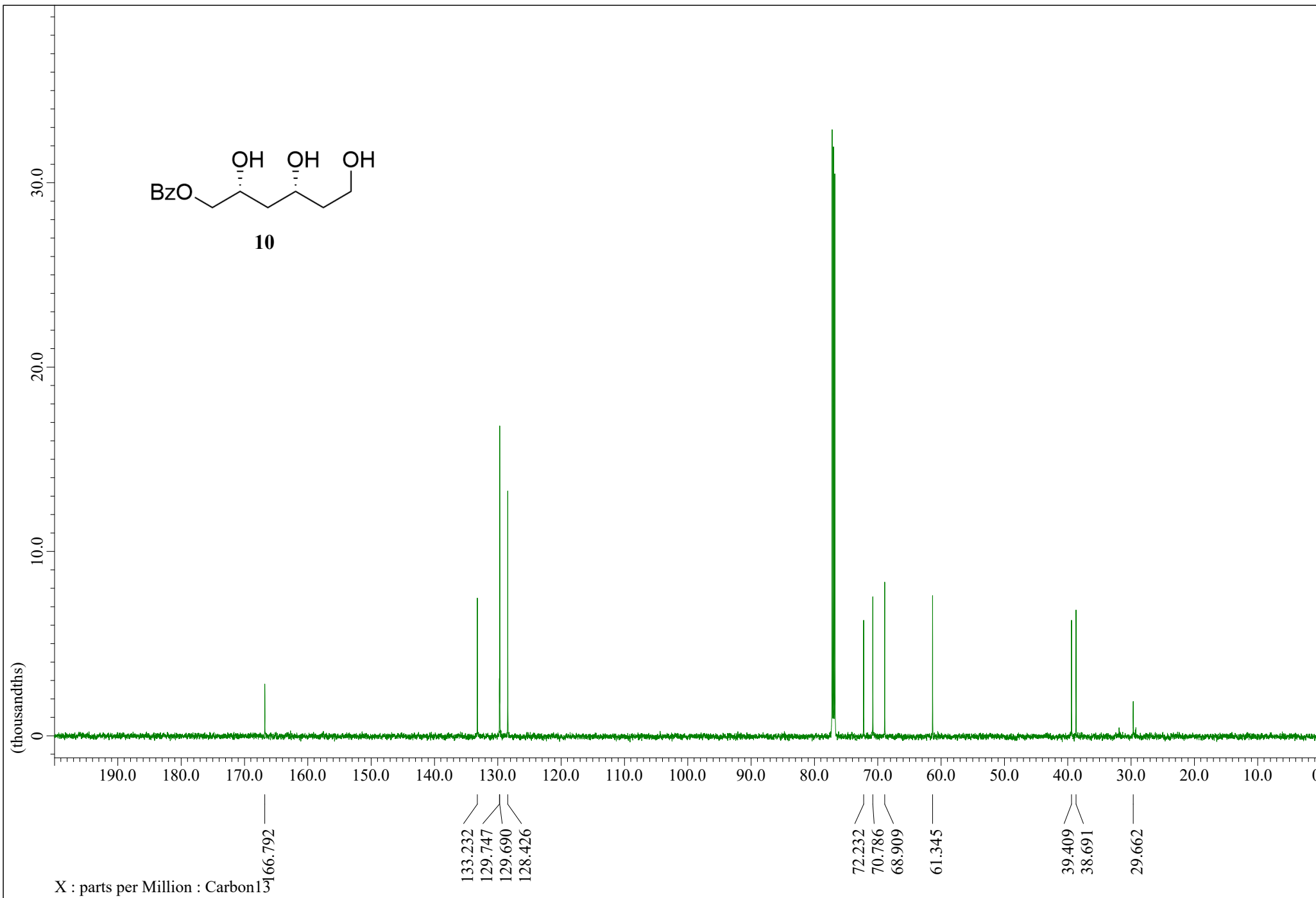


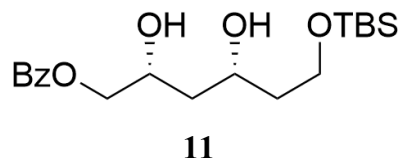
10



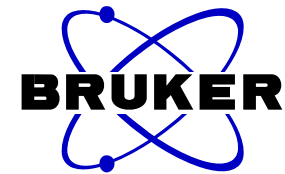
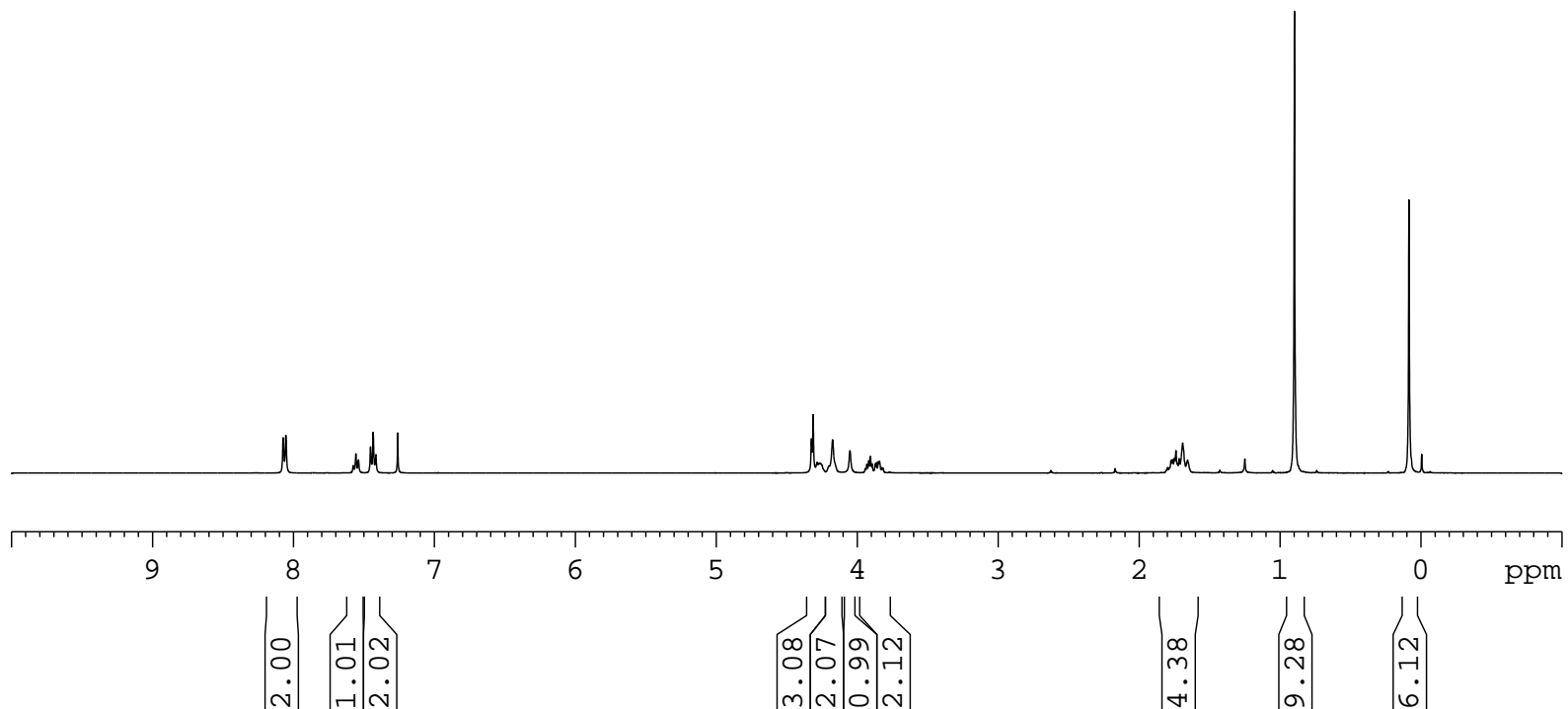


10





8.0724  
8.0540  
7.5754  
7.5570  
7.5385  
7.4532  
7.4338  
7.4149  
7.2600  
4.3246  
4.3125  
4.2846  
4.2702  
4.2601  
4.2010  
4.1742  
4.0511  
3.9429  
3.9313  
3.9180  
3.9056  
3.8941  
3.8725  
3.8634  
3.8500  
3.8412  
3.8249  
3.8158  
1.7730  
1.7624  
1.7490  
1.7383  
1.7275  
1.7153  
1.6906  
1.6554  
0.8975  
0.0848



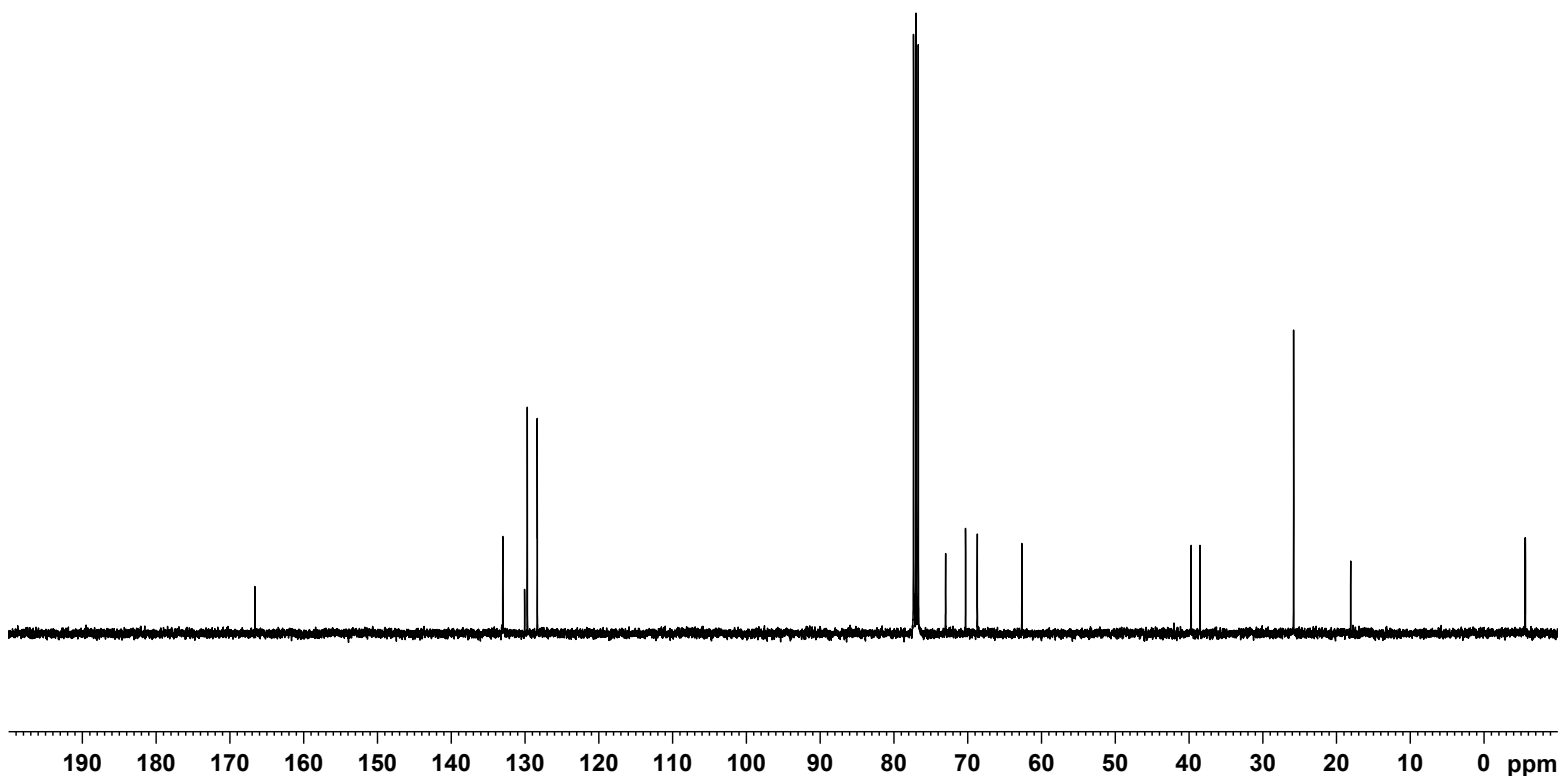
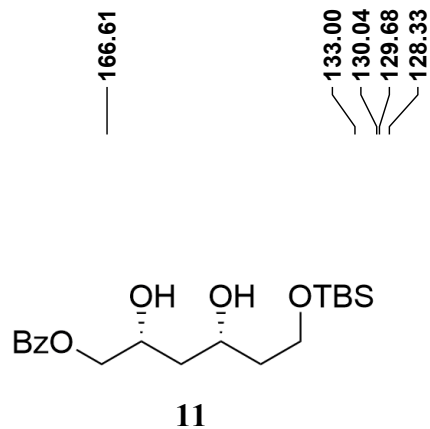
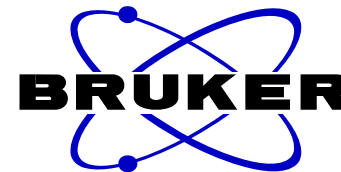
```

NAME      FJJ171121-1
EXPNO     1
PROCNO    1
Date_     20171122
Time      17.07
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        90.5
DW        60.800 usec
DE        6.50 usec
TE        296.3 K
D1        1.00000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        15.50 usec
PL1       -1.00 dB
PL1W      13.18669796 W
SF01      400.1724712 MHz
SI        32768
SF        400.1700028 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



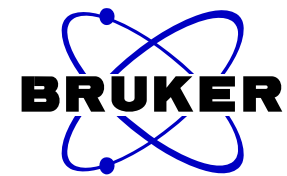
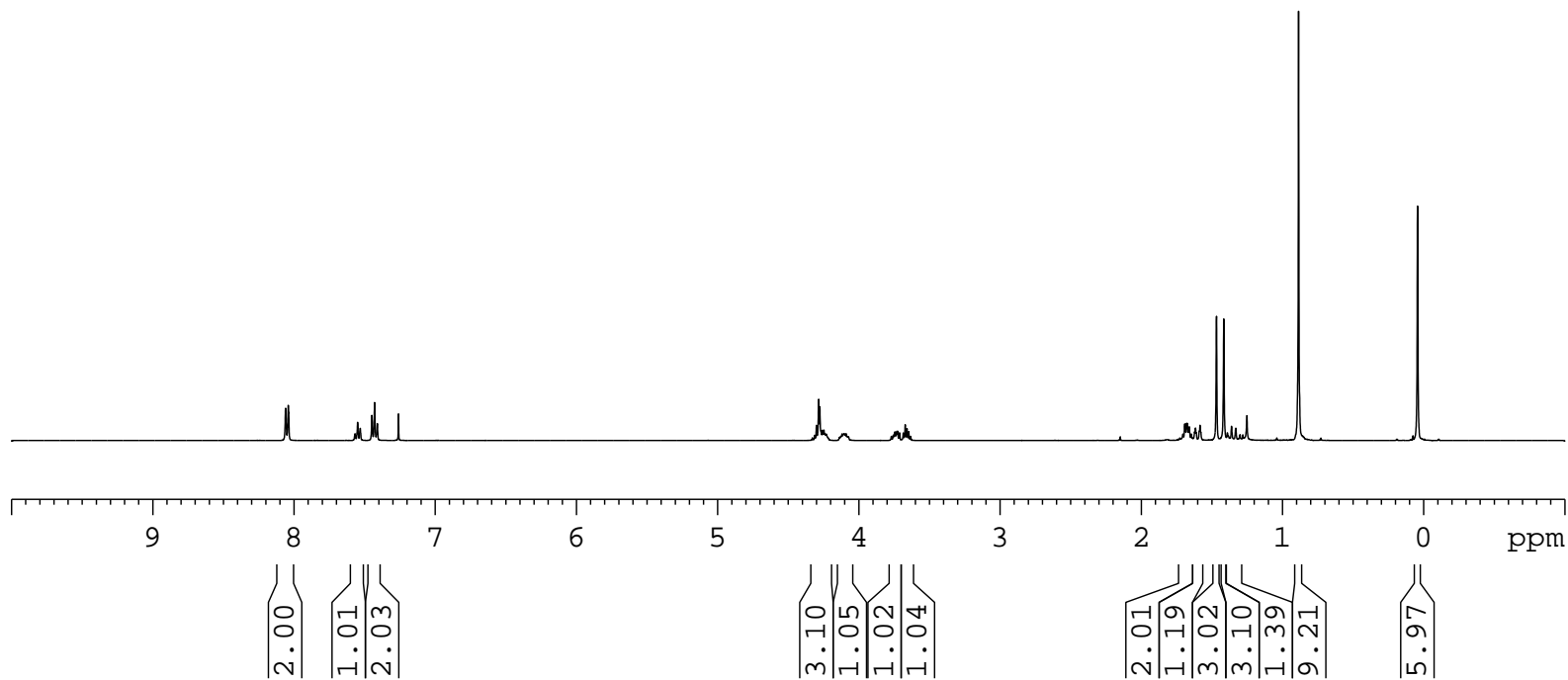
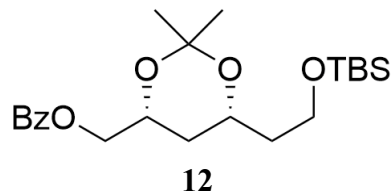


NAME FJJ171121-1-C13  
EXPNO 1  
PROCNO 1  
Date\_ 20171122  
Time 17.24  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 205  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.5 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 57.32743073 W  
SFO1 100.6328888 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.26 dB  
PL13 14.46 dB  
PL2W 13.18669796 W  
PL12W 0.49446553 W  
PL13W 0.37509048 W  
SFO2 400.1716007 MHz  
SI 32768  
SF 100.6228282 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

8.0587  
8.0406  
8.0374  
7.5492  
7.5306  
7.4482  
7.4288  
7.4099  
4.2999  
4.2846  
4.2758  
4.2577  
4.2471  
4.2405  
4.1187  
4.1075  
4.1013  
4.0900  
3.7493  
3.7442  
3.7361  
3.7310  
3.7245  
3.7112  
3.6847  
3.6713  
3.6586  
3.6460  
1.6917  
1.6783  
1.6730  
1.6648  
1.6593  
1.6216  
1.6153  
1.6097  
1.5889  
1.5834  
1.5781  
1.4670  
1.4151  
1.3894  
1.3604  
1.3300  
0.8854  
0.0429

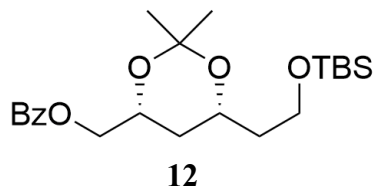


```

NAME          FJJ1711113
EXPNO         1
PROCNO        1
Date_         20171113
Time          11.06
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            32
DW            60.800 usec
DE            6.50 usec
TE            296.7 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             15.50 usec
PL1            -1.00 dB
PL1W          13.18669796 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700028 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
  
```



— 166.34

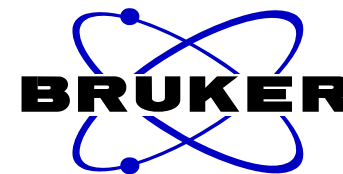
132.93  
130.04  
129.63  
128.27

— 98.71

77.32  
77.00  
76.68  
67.54  
67.41  
65.20  
58.64

— 39.37  
33.55  
30.05  
25.86  
19.73  
18.21

— -5.42



```

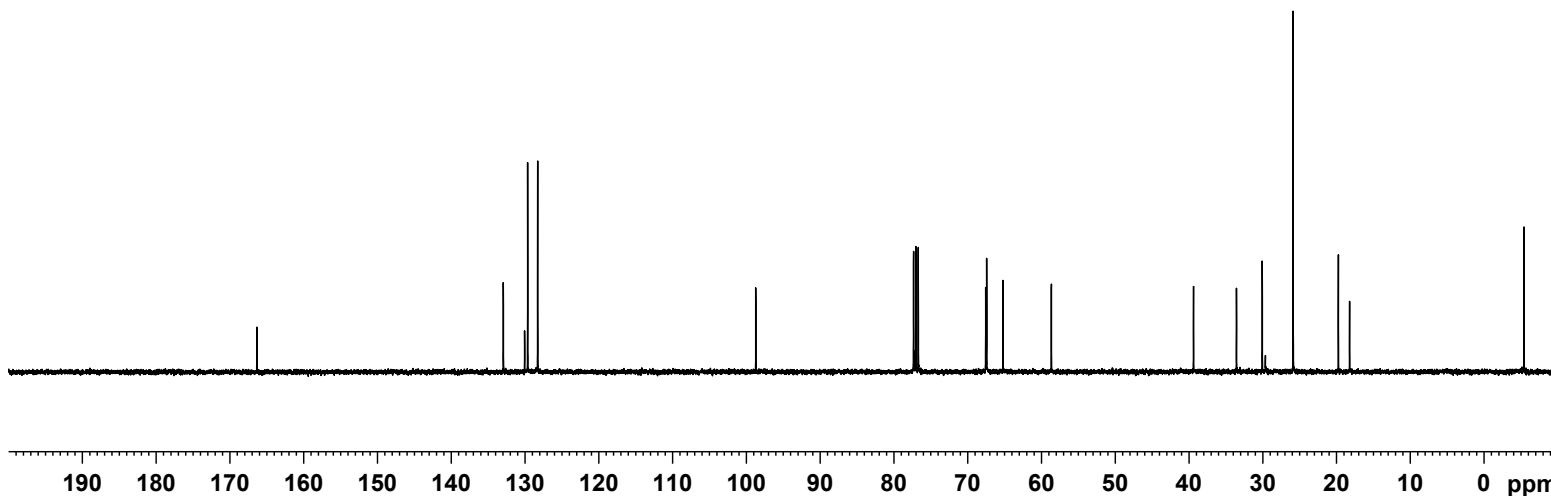
NAME      FJJ171113-C13
EXPNO     1
PROCNO    1
Date_     20171113
Time      11.13
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         57
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         297.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

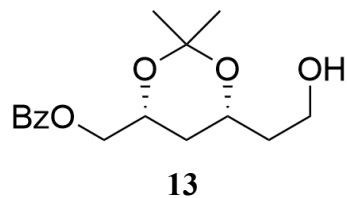
```

===== CHANNEL f1 =====
NUC1      13C
P1         8.50 usec
PL1        -2.00 dB
PL1W      57.32743073 W
SFO1      100.6328888 MHz
  
```

```

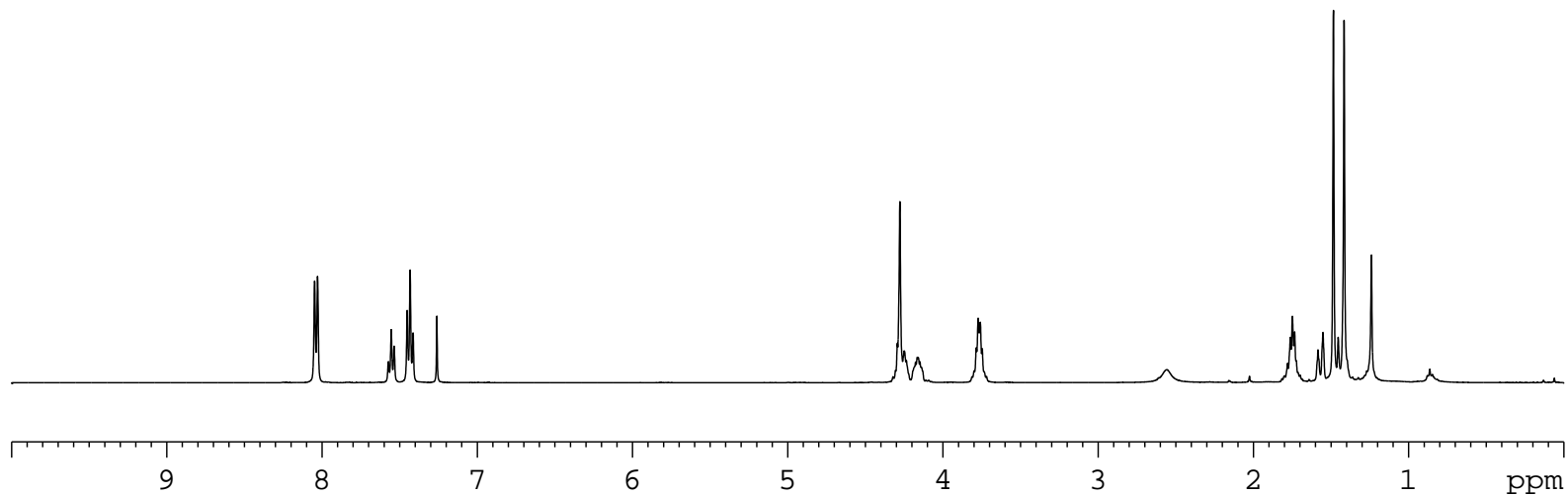
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       13.26 dB
PL13       14.46 dB
PL2W      13.18669796 W
PL12W     0.49446553 W
PL13W     0.37509048 W
SFO2      400.1716007 MHz
SI         32768
SF         100.6228318 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```





8.0474  
8.0284  
7.5717  
7.5534  
7.5350  
7.4509  
7.4318  
7.4128  
7.2597

4.2932  
4.2771  
4.2492  
4.2350  
4.1757  
4.1648  
4.1580  
4.1474  
3.7848  
3.7729  
3.7590  
3.7465  
2.5586  
1.7796  
1.7613  
1.7485  
1.7343  
1.7236  
1.5833  
1.5514  
1.4840  
1.4151



2.00  
1.00  
2.01

3.12  
1.06  
2.04

0.96  
2.16  
1.12  
3.01  
0.49  
3.25

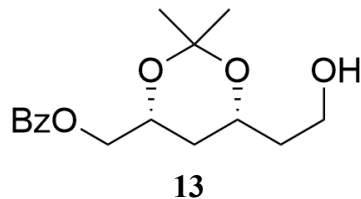


```

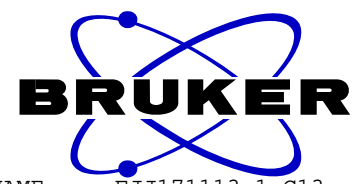
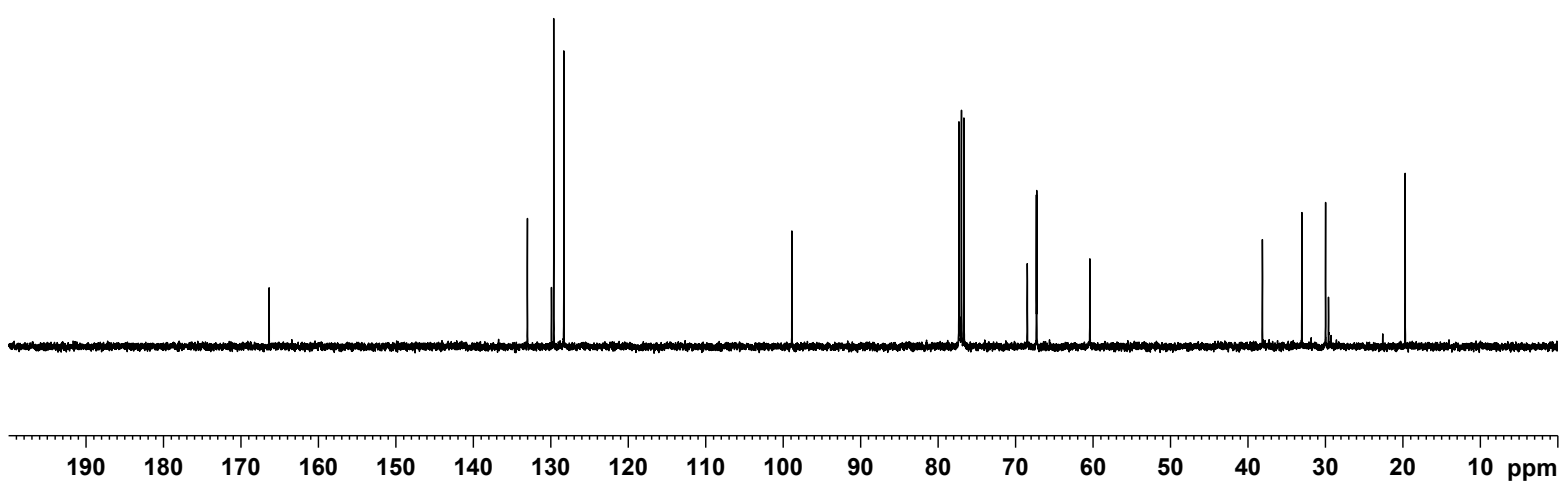
NAME      FJJ171113-1
EXPNO     1
PROCNO    1
Date_     20171114
Time      9.43
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG         57
DW        60.800 usec
DE         6.50 usec
TE        295.1 K
D1         1.0000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        15.50 usec
PL1       -1.00 dB
PL1W      13.18669796 W
SF01      400.1724712 MHz
SI        32768
SF        400.1700028 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB         0
PC        1.00
  
```



166.35  
 133.01  
 129.91  
 129.61  
 128.31  
 98.87  
 77.32  
 77.20  
 77.00  
 76.68  
 68.50  
 67.33  
 67.27  
 60.42  
 38.15  
 33.05  
 29.99  
 29.61  
 19.71



```

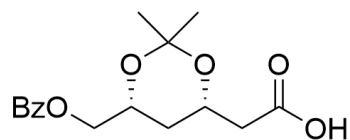
NAME      FJJ171113-1-C13
EXPNO     1
PROCNO    1
Date_     20171114
Time      9.53
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS        76
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        203
DW        20.800 usec
DE        6.50 usec
TE        296.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        8.50 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6328888 MHz
  
```

```

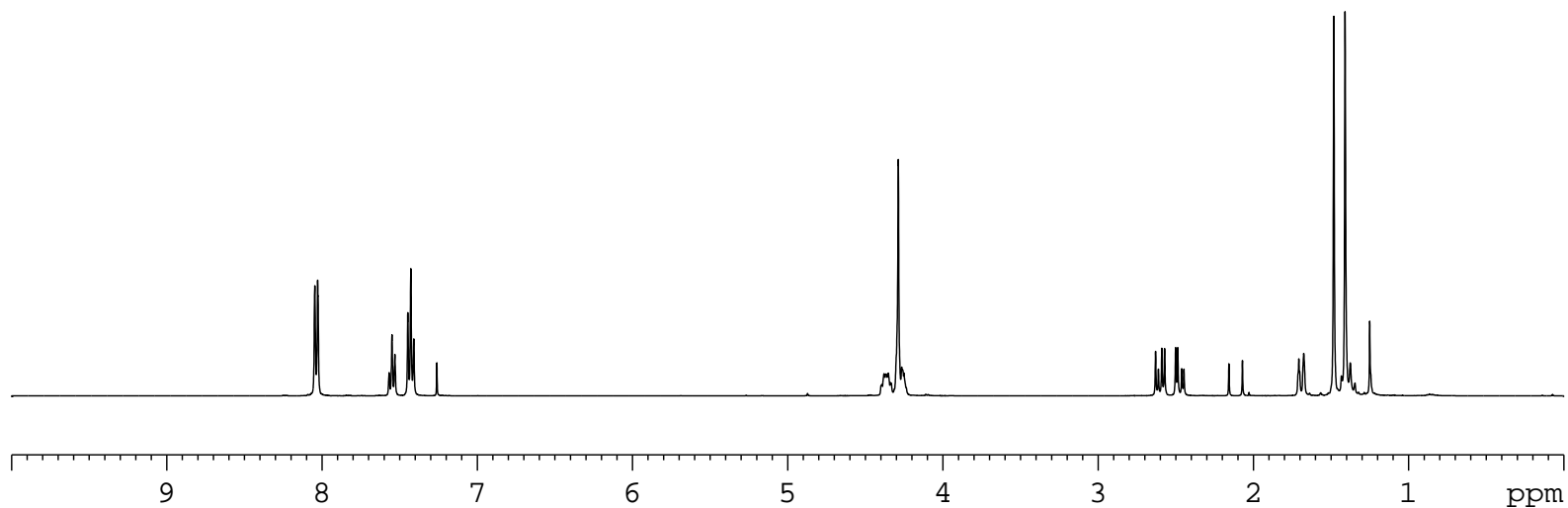
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -1.00 dB
PL12      13.26 dB
PL13      14.46 dB
PL2W      13.18669796 W
PL12W     0.49446553 W
PL13W     0.37509048 W
SFO2      400.1716007 MHz
SI        32768
SF        100.6228333 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



14

8.0460  
8.0279  
8.0254  
7.5676  
7.5491  
7.5306  
7.4469  
7.4275  
7.4086  
7.2598

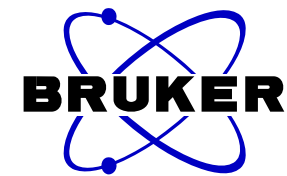
4.3930  
4.3795  
4.3663  
4.3514  
4.3381  
4.3324  
4.2873  
4.2655  
4.2599  
4.2503  
2.6297  
2.6114  
2.5896  
2.5715  
2.5007  
2.4868  
2.4609  
2.4469  
2.1574  
2.0702  
1.7068  
1.7017  
1.6752  
1.4816  
1.4326  
1.4089



2.00  
1.03  
2.07

1.07  
3.05

1.13  
1.01  
0.17  
0.18  
1.01  
2.98  
0.23  
3.29  
0.63



NAME fjj171114  
EXPNO 1  
PROCNO 1  
Date\_ 20171114  
Time 19.37  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 36  
DW 60.800 usec  
DE 6.50 usec  
TE 295.6 K  
D1 1.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 15.50 usec  
PL1 -1.00 dB  
PL1W 13.18669796 W  
SF01 400.1724712 MHz  
SI 32768  
SF 400.1700030 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

— 176.21

— 166.36

— 133.02

— 129.81

— 129.60

— 128.29

— 99.16

— 77.32

— 77.00

— 76.68

— 67.20

— 67.11

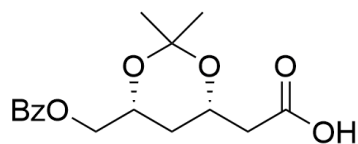
— 65.27

— 41.02

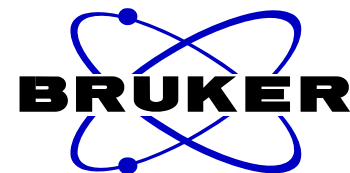
— 32.57

— 29.74

— 19.51



14



```

NAME      fjj171114-C13
EXPNO     1
PROCNO    1
Date_     20171114
Time      19.48
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         42
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

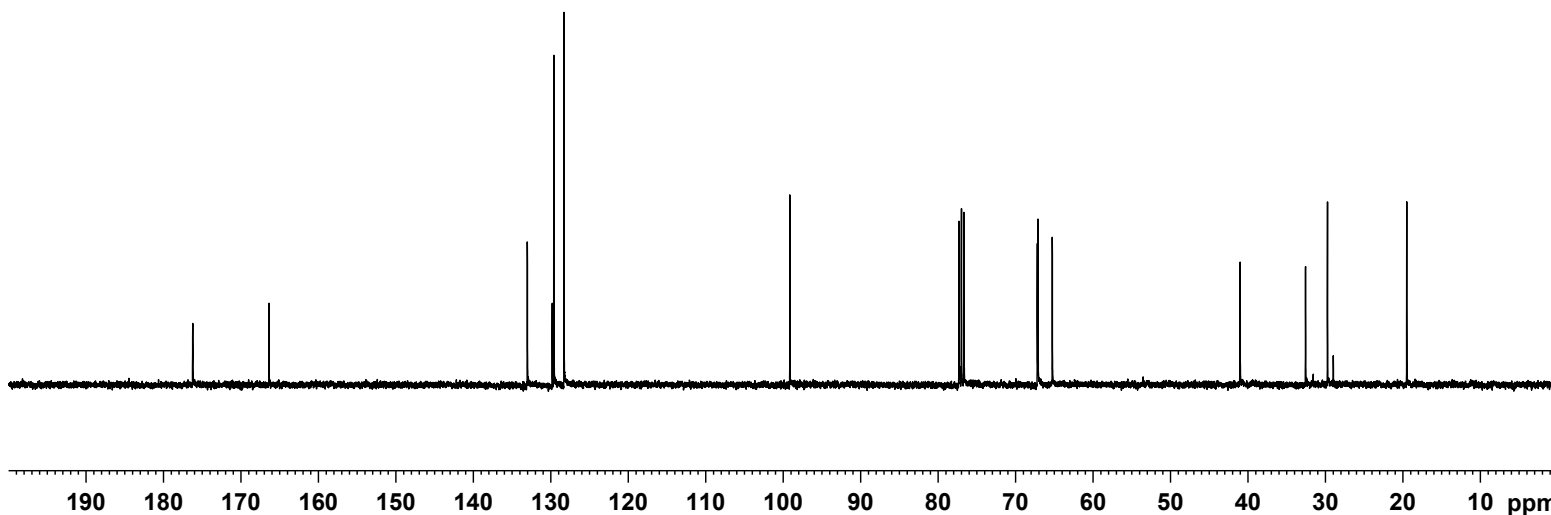
===== CHANNEL f1 =====
NUC1      13C
P1        8.50 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6328888 MHz

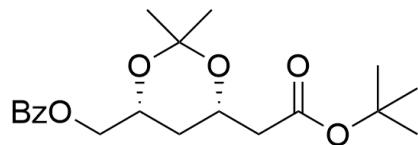
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -1.00 dB
PL12      13.26 dB
PL13      14.46 dB
PL2W      13.18669796 W
PL12W     0.49446553 W
PL13W     0.37509048 W
SFO2      400.1716007 MHz
SI        32768
SF        100.6228370 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

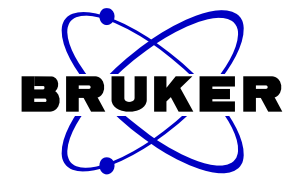




15

8.0365  
8.0180  
7.5573  
7.5388  
7.5203  
7.4355  
7.4162  
7.3972

4.3165  
4.2857  
4.2693  
4.2426  
4.2389  
4.2280  
2.4719  
2.4541  
2.4341  
2.4164  
2.3515  
2.3366  
2.3136  
2.2987  
1.6653  
1.6601  
1.6337  
1.4692  
1.4243  
1.3890  
1.3715  
1.3418  
1.3120

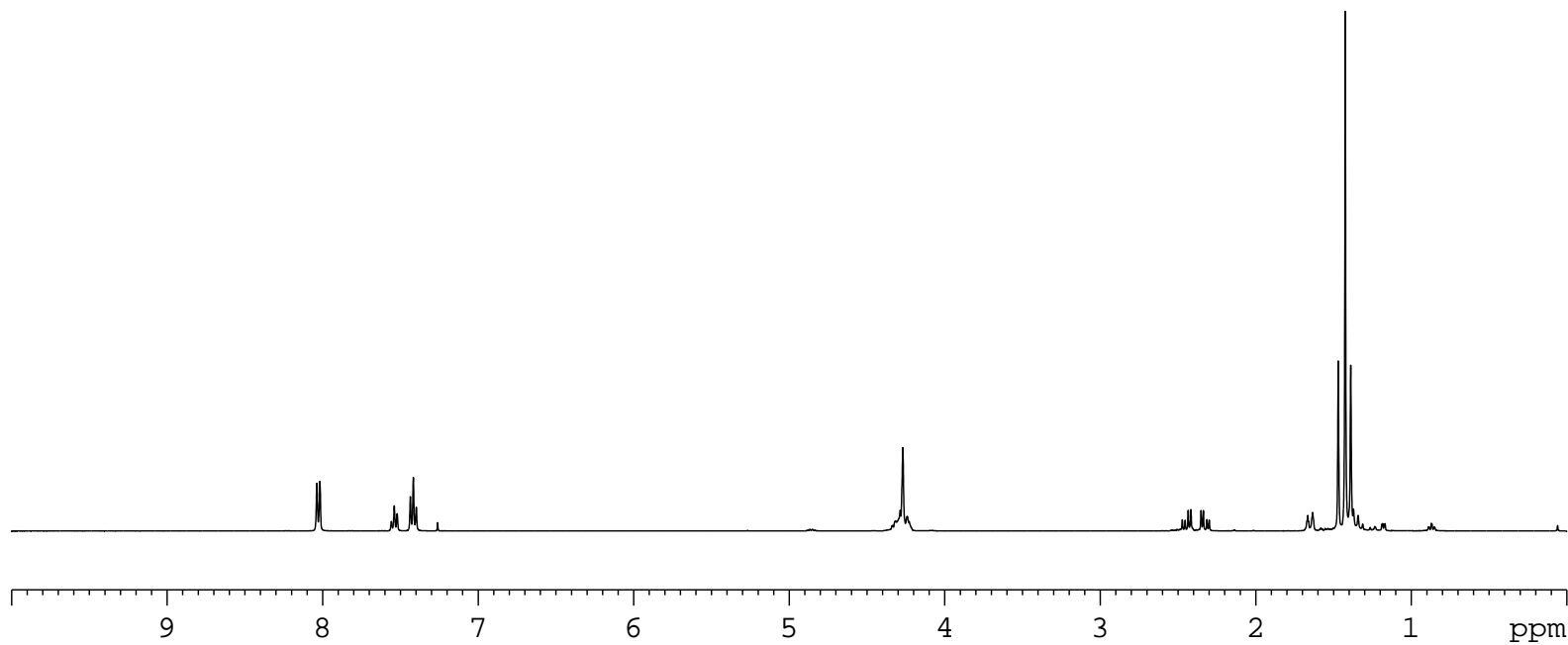


```

NAME          FJJ171115
EXPNO         1
PROCNO       1
Date_        20171115
Time         20.40
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           2
SWH          8223.685 Hz
FIDRES       0.125483 Hz
AQ           3.9846387 sec
RG           36
DW           60.800 usec
DE           6.50 usec
TE           295.6 K
D1           1.0000000 sec
TD0          1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            15.50 usec
PL1           -1.00 dB
PL1W         13.18669796 W
SF01         400.1724712 MHz
SI           32768
SF           400.1700025 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



2.00  
1.01  
2.05  
1.18  
2.15  
0.73  
2.02  
1.08  
3.18  
8.31  
2.91  
1.10



169.97  
166.27

132.95  
129.93  
129.59  
128.27

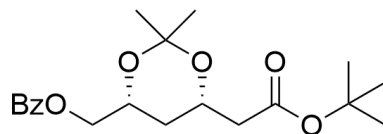
98.89

80.58  
77.32  
77.00  
76.69  
67.28  
67.19  
65.80

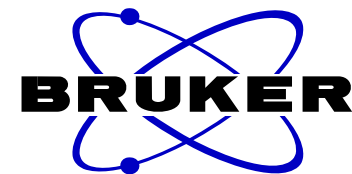
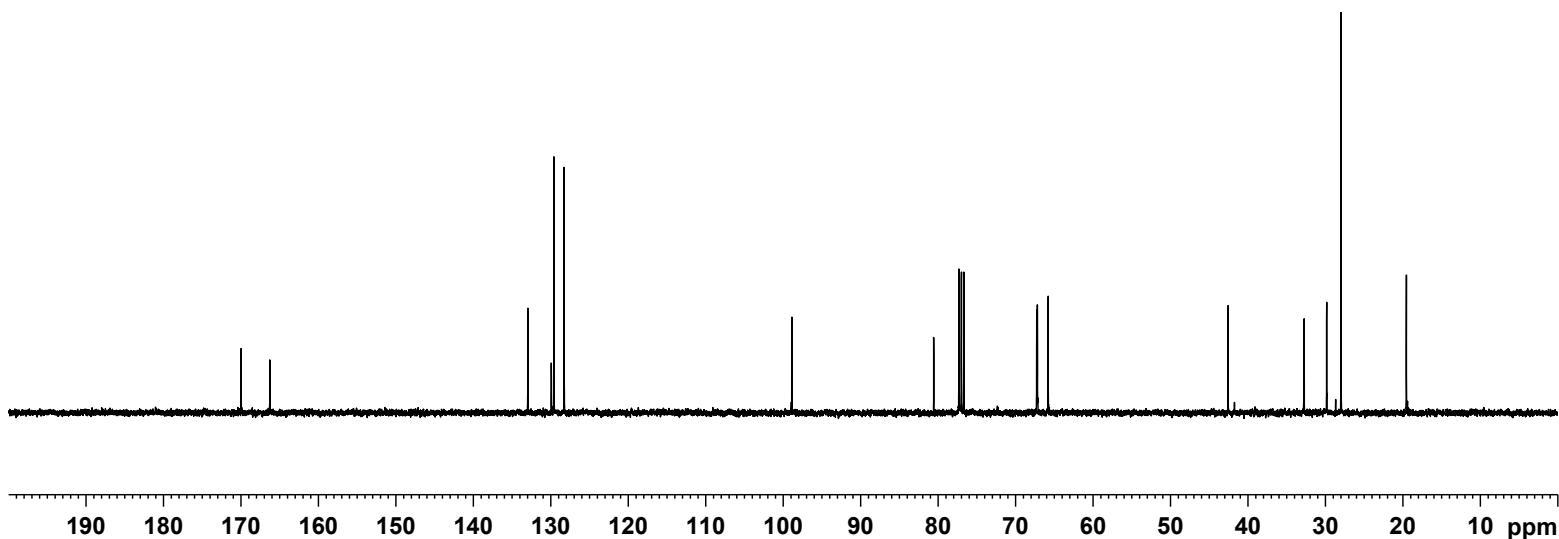
42.57

32.75  
29.85  
27.99

19.56



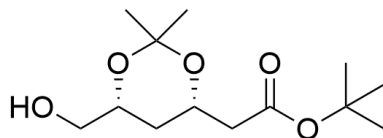
15



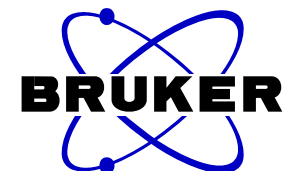
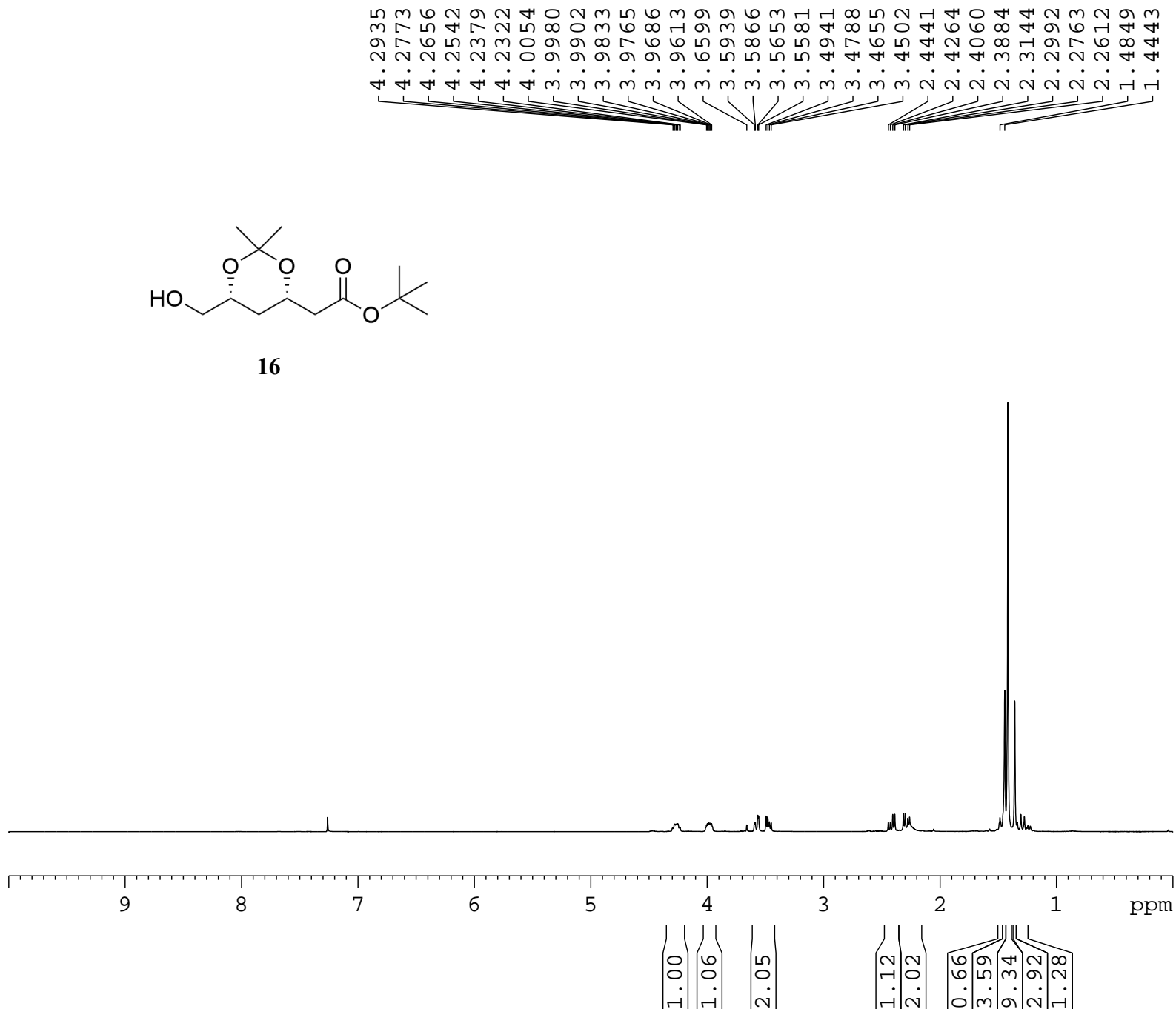
NAME FJJ171115-C13  
EXPNO 1  
PROCNO 1  
Date\_ 20171115  
Time 20.48  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 36  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 296.1 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 57.32743073 W  
SFO1 100.6328888 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.26 dB  
PL13 14.46 dB  
PL2W 13.18669796 W  
PL12W 0.49446553 W  
PL13W 0.37509048 W  
SFO2 400.1716007 MHz  
SI 32768  
SF 100.6228341 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



16



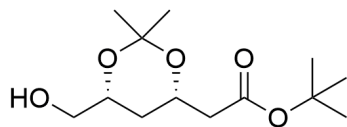
```

NAME      FJJ171116-1
EXPNO     1
PROCNO    1
Date_     20171118
Time      9.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG         64
DW         60.800 usec
DE         6.50 usec
TE         292.8 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        15.50 usec
PL1       -1.00 dB
PL1W      13.18669796 W
SF01      400.1724712 MHz
SI        32768
SF        400.1700028 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

— 170.12



16

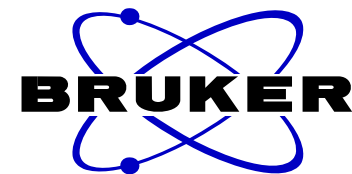
— 98.83

80.62  
77.32  
77.20  
77.00  
76.68  
69.47  
65.84  
65.69

— 42.57

31.76  
29.86  
28.01

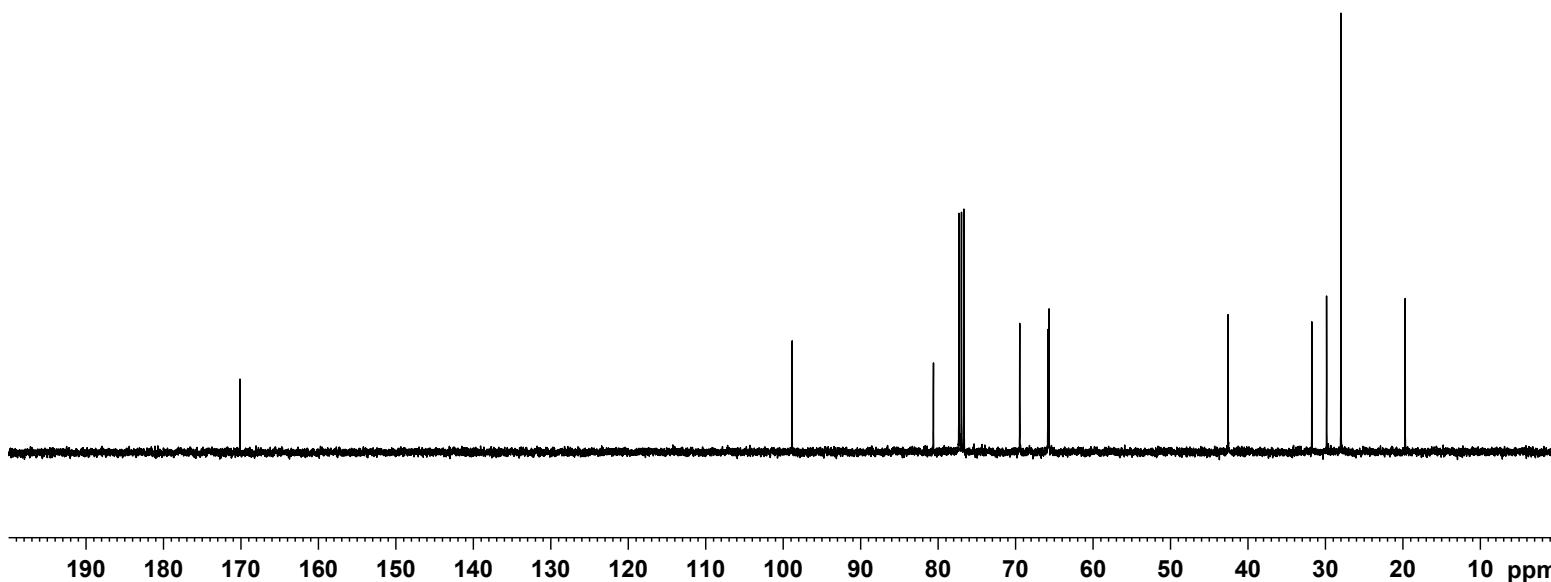
— 19.71



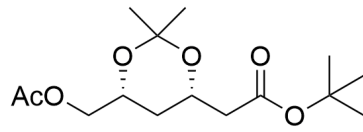
NAME FJJ171116-1-C13  
EXPNO 1  
PROCNO 1  
Date\_ 20171118  
Time 9.47  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 56  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 293.7 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 57.32743073 W  
SFO1 100.6328888 MHz

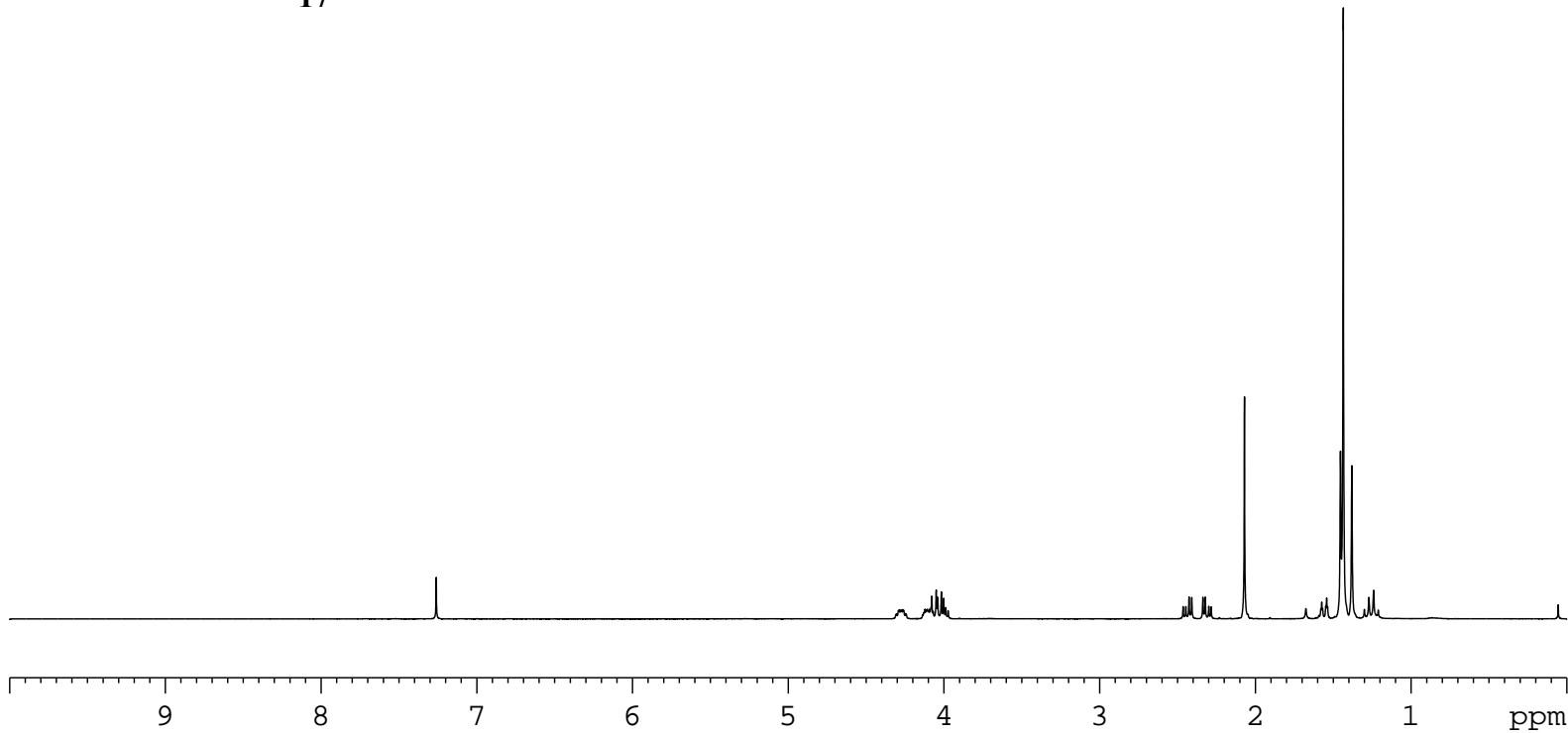
==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 14.26 dB  
PL13 14.46 dB  
PL2W 13.18669796 W  
PL12W 0.39276794 W  
PL13W 0.37509048 W  
SFO2 400.1716007 MHz  
SI 32768  
SF 100.6228318 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



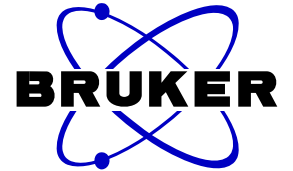
4.3025  
 4.2916  
 4.2867  
 4.2794  
 4.2748  
 4.2701  
 4.2637  
 4.2582  
 4.2470  
 4.2411  
 4.1272  
 4.1210  
 4.1118  
 4.1059  
 4.0983  
 4.0921  
 4.0775  
 4.0682  
 4.0492  
 4.0397  
 4.0162  
 4.0009  
 3.9878  
 3.9725  
 2.4636  
 2.4460  
 2.4256  
 2.4080  
 2.3366  
 2.3214  
 2.2986  
 2.2834  
 2.0697  
 1.5793  
 1.5735  
 1.5676  
 1.5478  
 1.5418  
 1.5359  
 1.4531  
 1.4352  
 1.3802  
 1.2996  
 1.2706  
 1.2396  
 1.2103



17



1.01  
 3.01  
 1.01  
 1.00  
 2.93  
 1.13  
 2.88  
 8.99  
 3.00  
 1.62

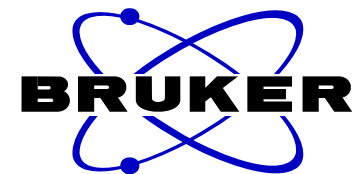


```

NAME          FJJ171218
EXPNO         1
PROCNO        1
Date_         20171218
Time          14.46
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            80.6
DW            60.800 usec
DE            6.50 usec
TE            294.6 K
D1            1.0000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            15.50 usec
PL1           -1.00 dB
PL1W         13.18669796 W
SF01         400.1724712 MHz
SI            32768
SF           400.1700028 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



NAME FJJ171218-C13  
EXPNO 1  
PROCNO 1  
Date\_ 20171218  
Time 14.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 81  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 203  
DW 20.800 usec  
DE 6.50 usec  
TE 294.8 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 57.32743073 W  
SFO1 100.6328888 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.26 dB  
PL13 14.46 dB  
PL2W 13.18669796 W  
PL12W 0.49446553 W  
PL13W 0.37509048 W  
SFO2 400.1716007 MHz  
SI 32768  
SF 100.6228289 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

170.93  
170.05

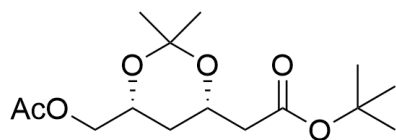
98.93

80.69

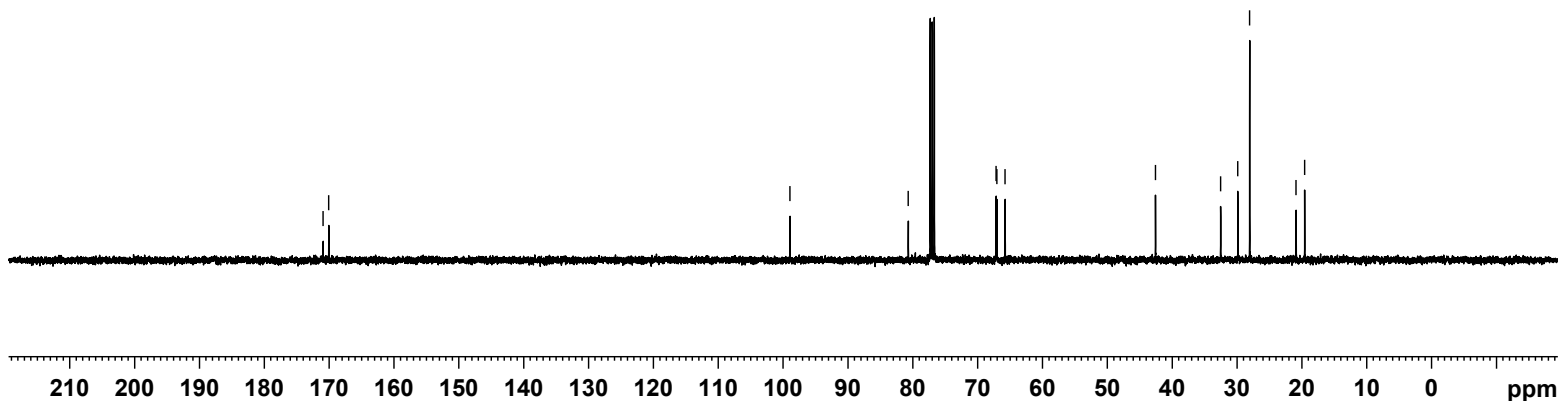
67.15  
67.03  
65.76

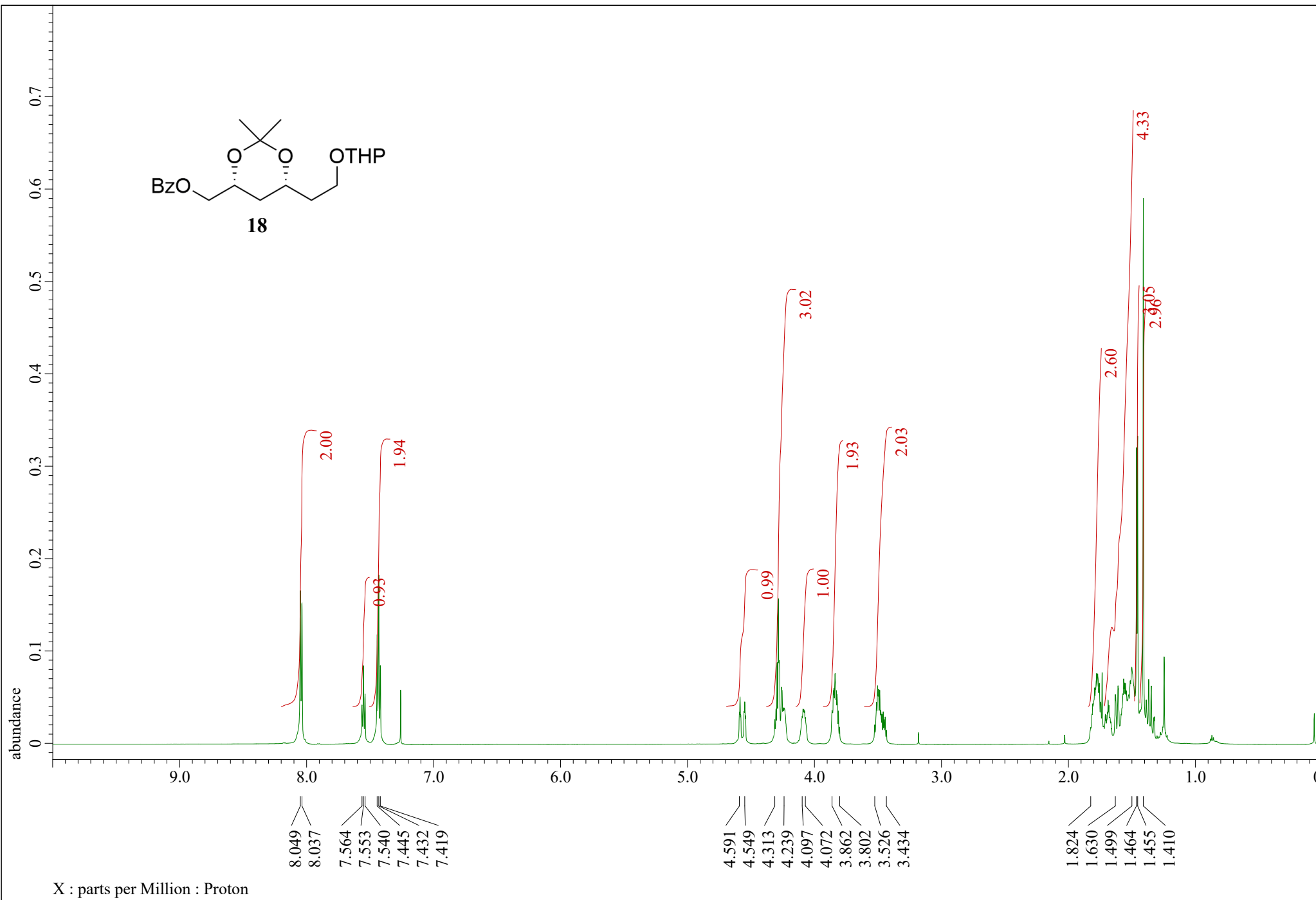
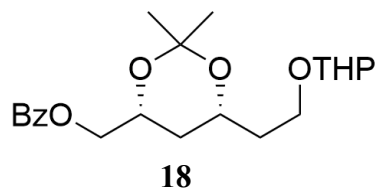
42.57

32.53  
29.88  
28.05  
20.88  
19.57

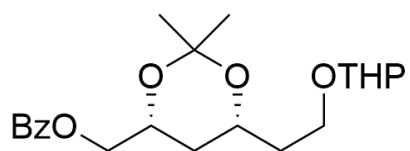


17

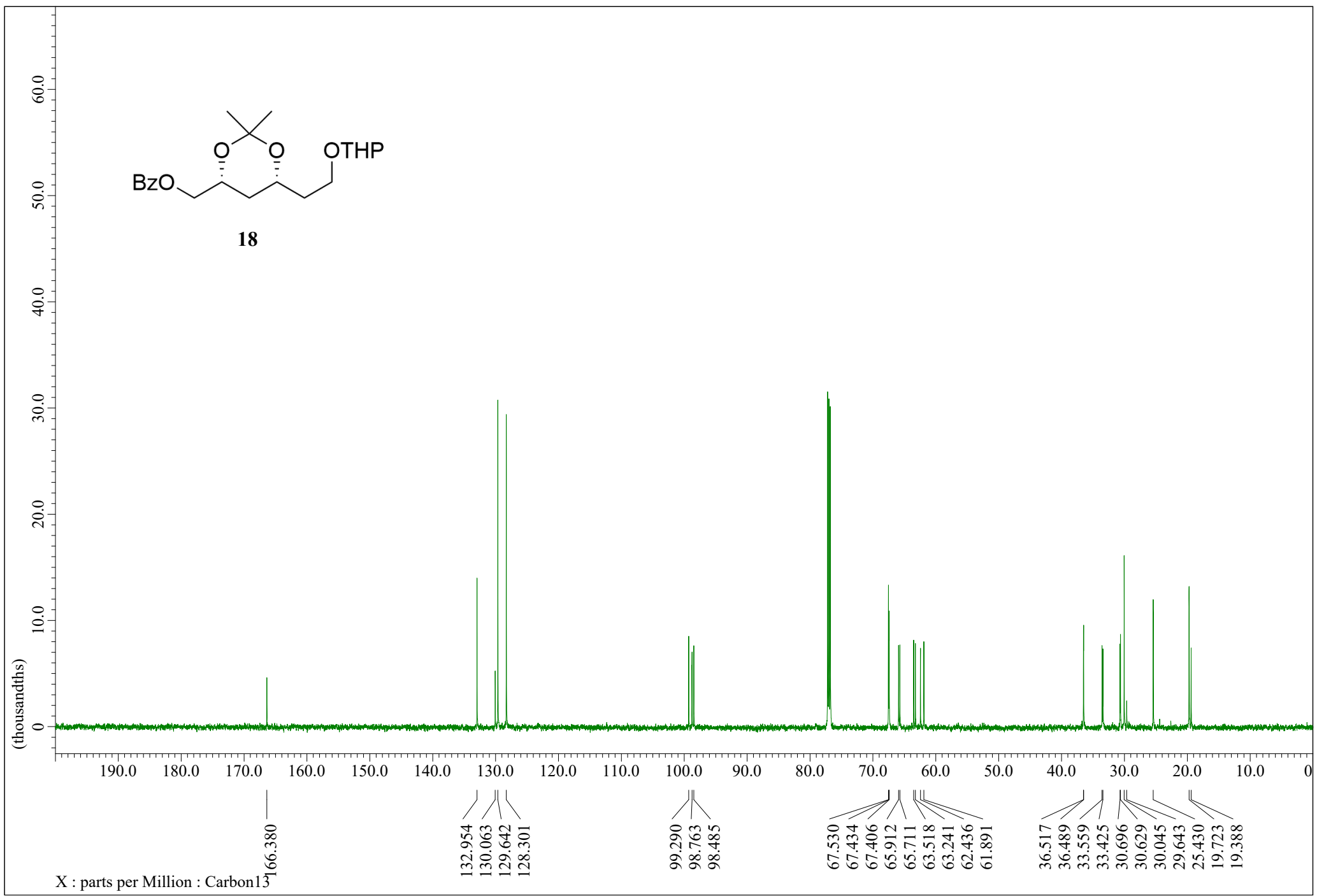


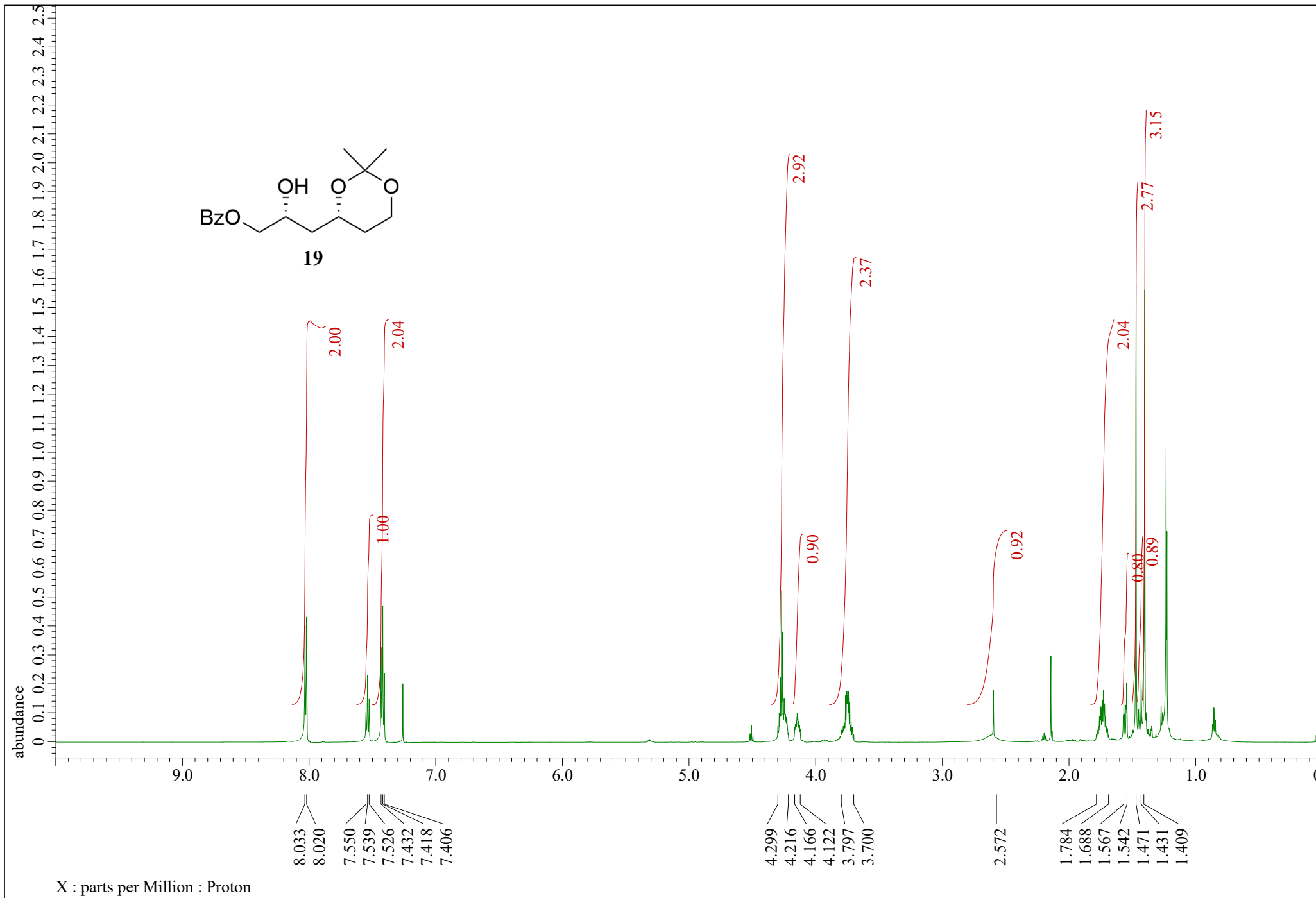


X : parts per Million : Proton

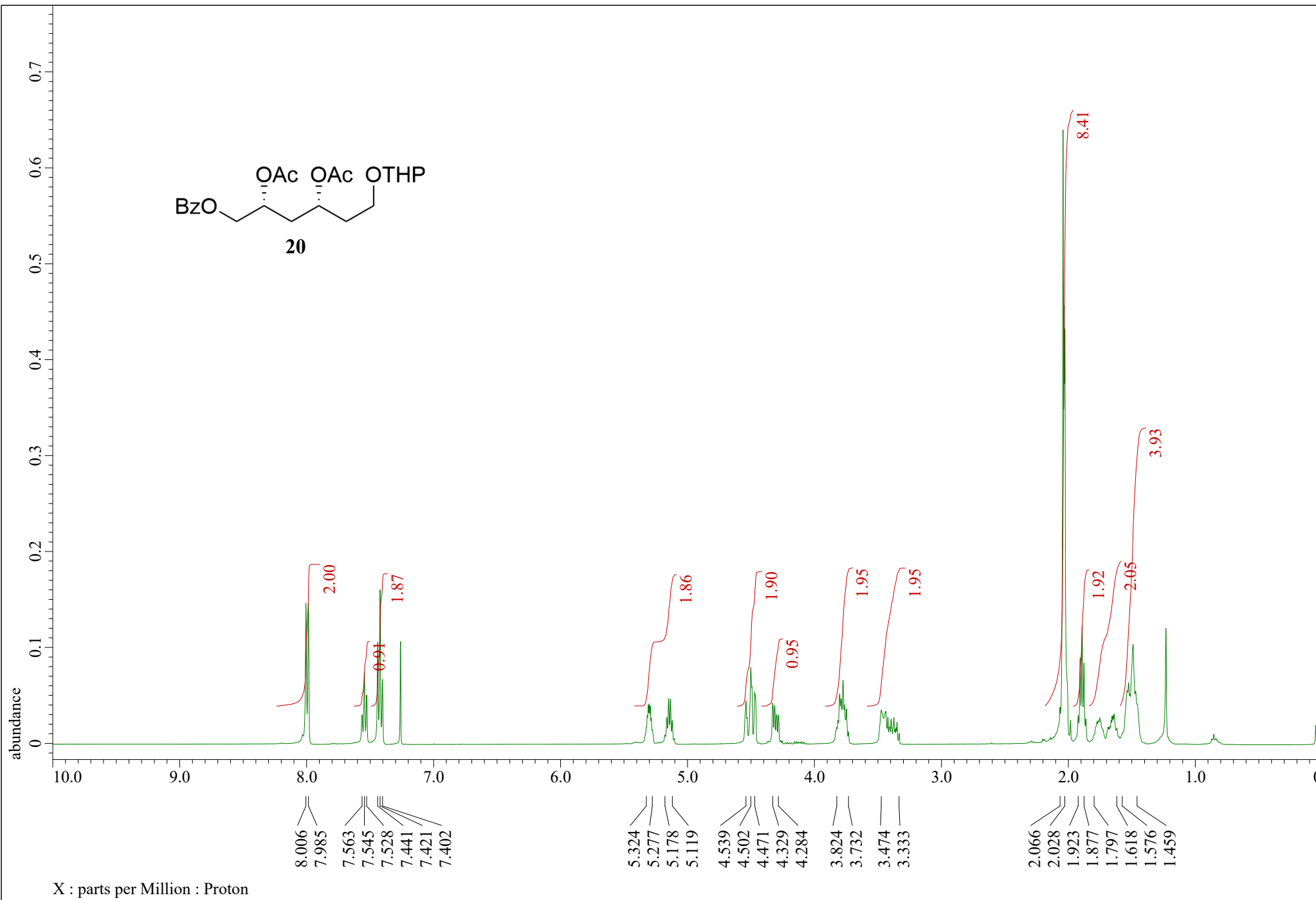
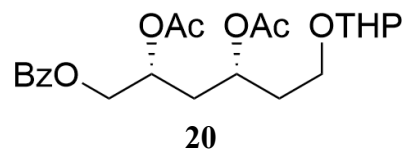


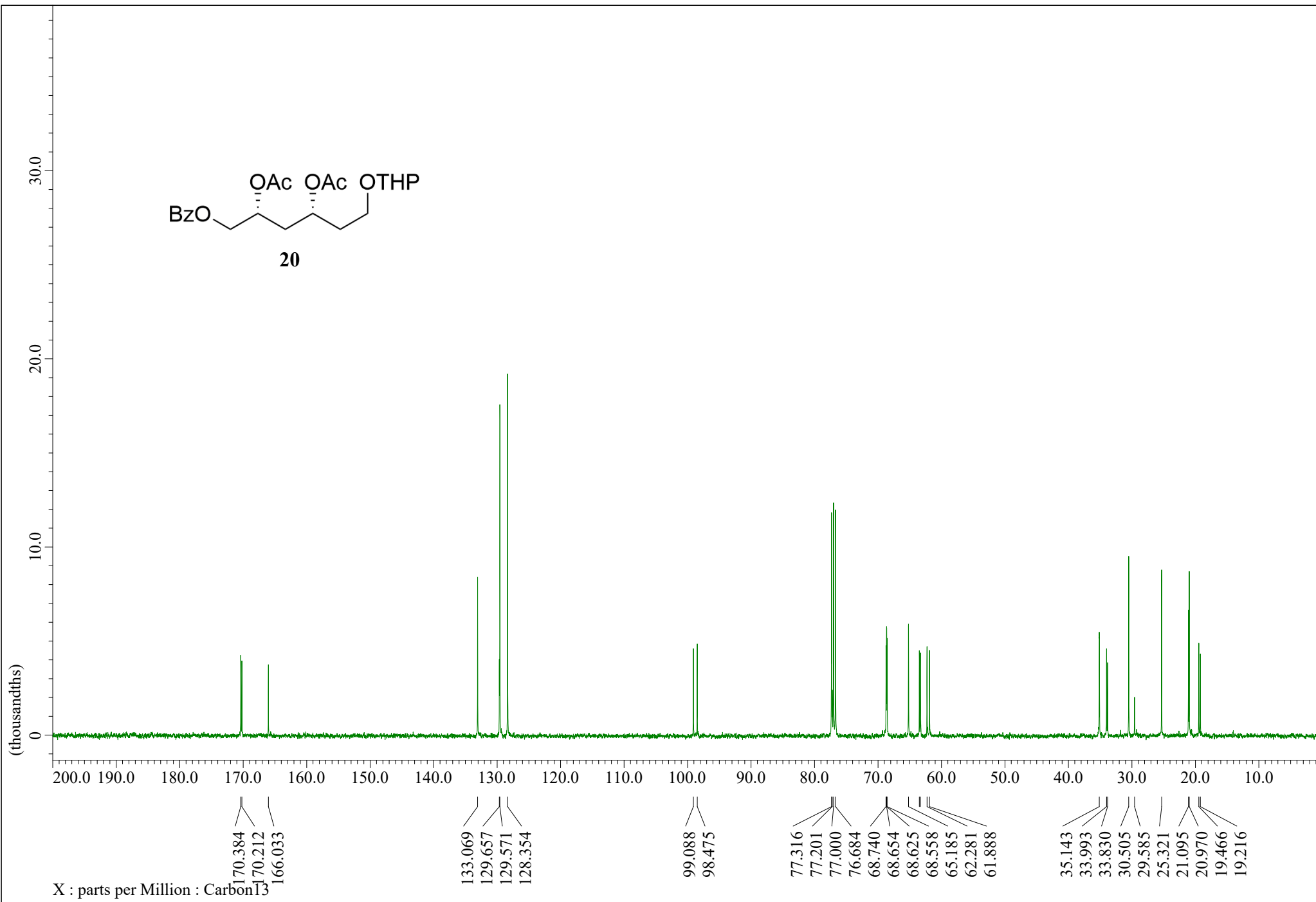
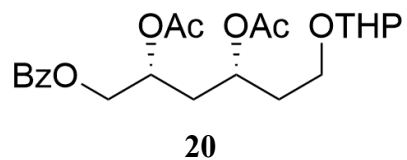
18

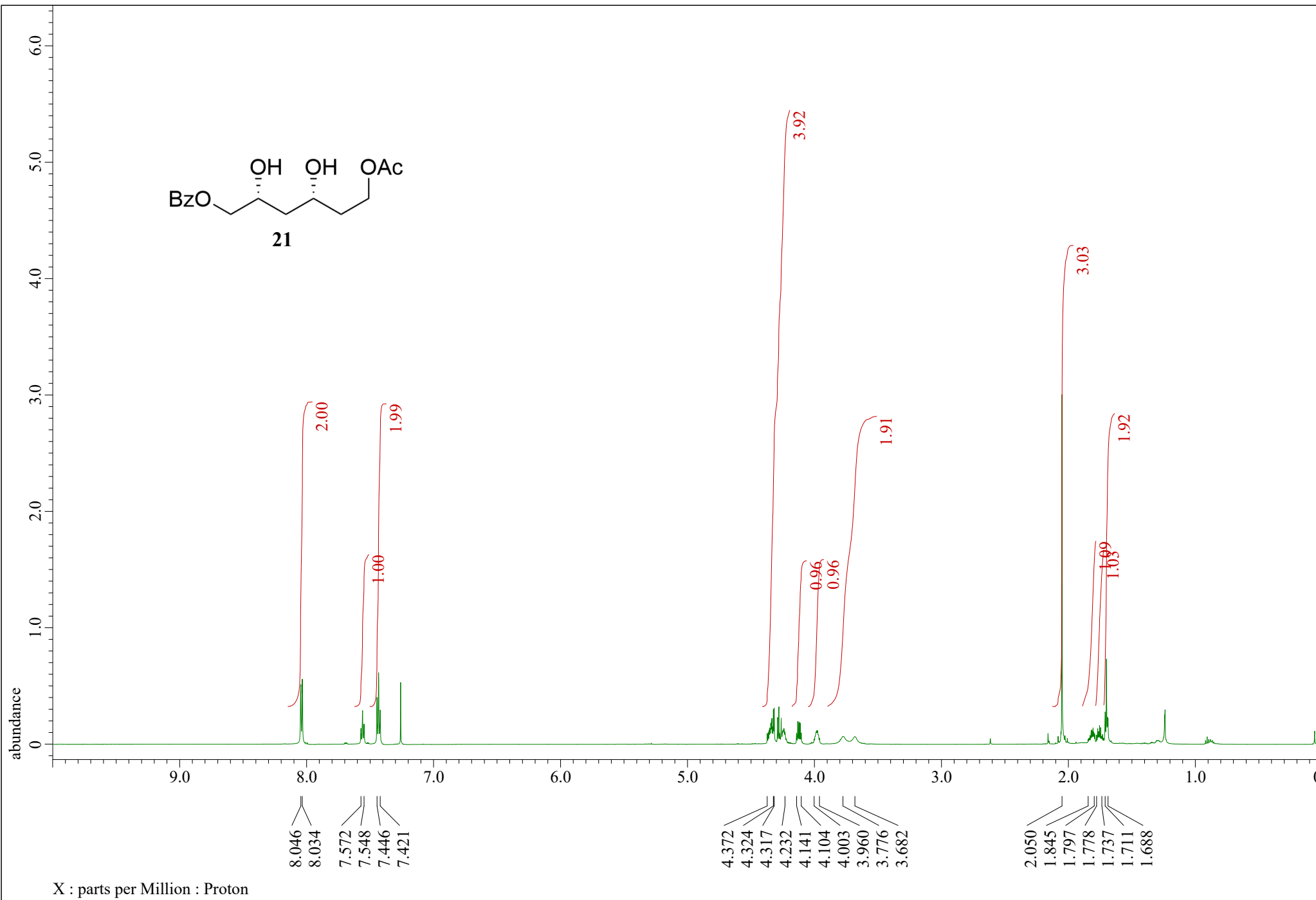
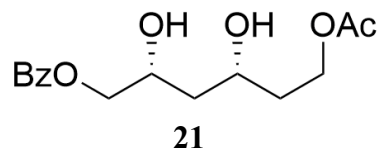




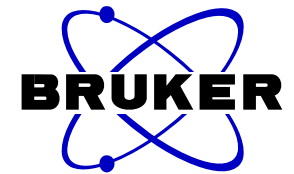
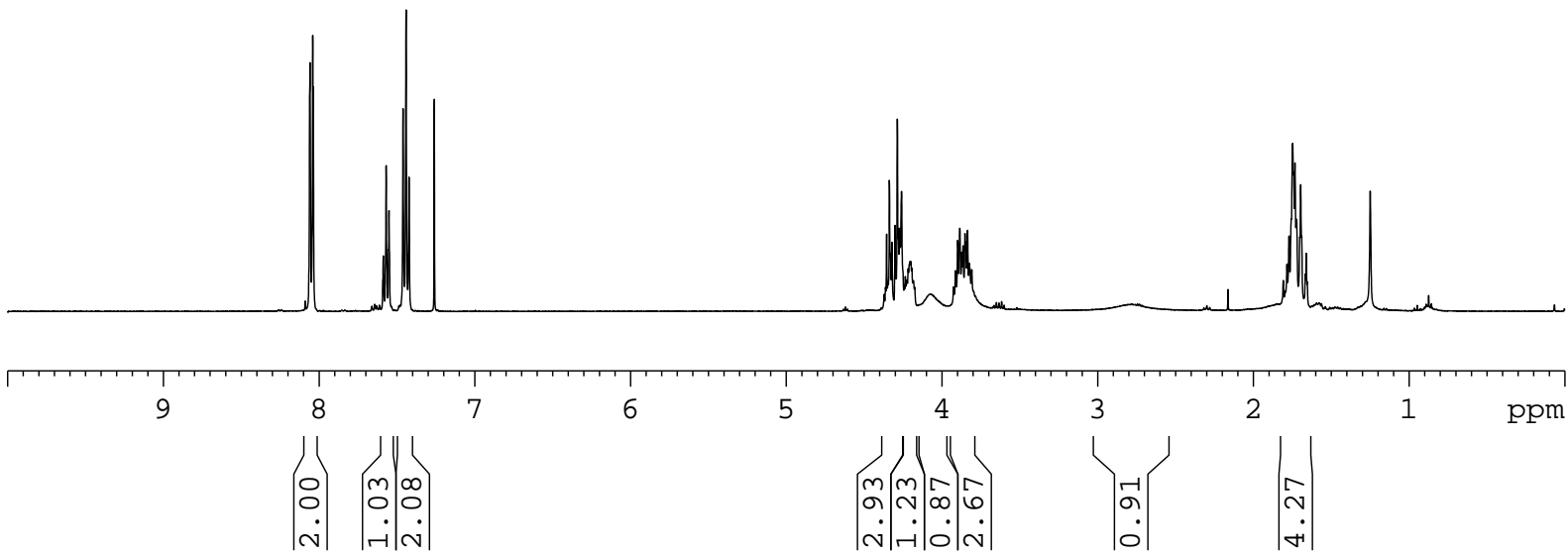
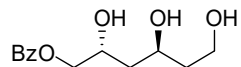








8.0585  
8.0407  
8.0373  
7.5880  
7.5694  
7.5509  
7.4611  
7.4414  
7.4225  
4.3556  
4.3374  
4.3308  
4.3217  
4.3014  
4.2864  
4.2801  
4.2735  
4.2684  
4.2596  
4.2352  
4.2169  
4.2100  
4.2051  
4.1997  
4.1936  
3.9126  
3.8987  
3.8853  
3.8727  
3.8646  
3.8523  
3.8472  
3.8357  
3.8250  
3.8083  
1.7845  
1.7713  
1.7479  
1.7417  
1.7333  
1.7232  
1.7020  
1.6963  
1.6902  
1.6668  
1.6607

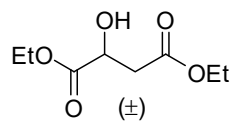


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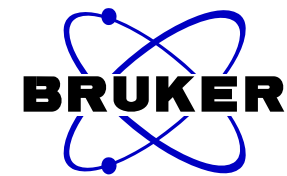
NAME      FJJ170717-2
EXPNO     1
PROCNO    1
Date_     20170722
Time      9.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        161
DW        60.800 usec
DE        6.50 usec
TE        298.7 K
D1        1.00000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        15.50 usec
PL1       -1.00 dB
PL1W     13.18669796 W
SF01     400.1724712 MHz
SI        32768
SF        400.1700030 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

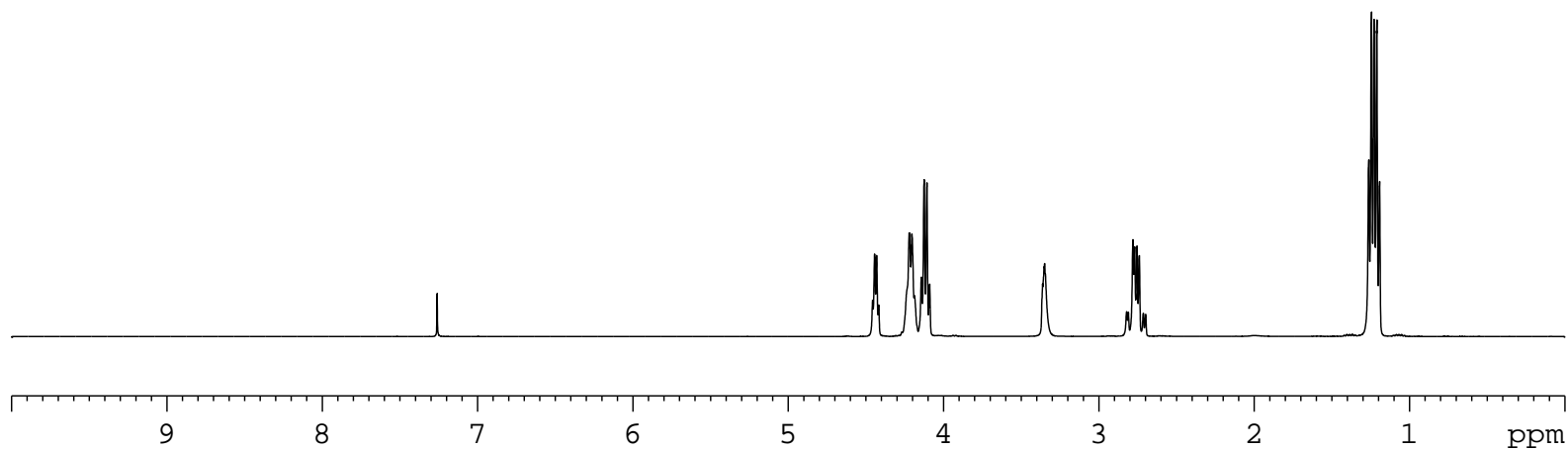


4.4566  
4.4437  
4.4308  
4.4172  
4.2213  
4.2191  
4.2037  
4.1865  
4.1423  
4.1245  
4.1068  
4.0891  
3.3622  
3.3531  
3.3491  
2.8211  
2.8099  
2.7804  
2.7692  
2.7543  
2.7393  
2.7134  
2.6984  
1.2622  
1.2461  
1.2444  
1.2279  
1.2116

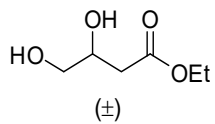


NAME FJJ171128-YUAN  
EXPNO 1  
PROCNO 1  
Date\_ 20171129  
Time 16.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 28.5  
DW 60.800 usec  
DE 6.50 usec  
TE 294.6 K  
D1 1.0000000 sec  
TD0 1

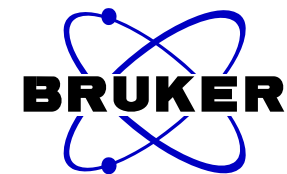
==== CHANNEL f1 =====  
NUC1 1H  
P1 15.50 usec  
PL1 -1.00 dB  
PL1W 13.18669796 W  
SF01 400.1724712 MHz  
SI 32768  
SF 400.1700025 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



1.00  
1.99  
2.03  
0.98  
2.06  
6.08

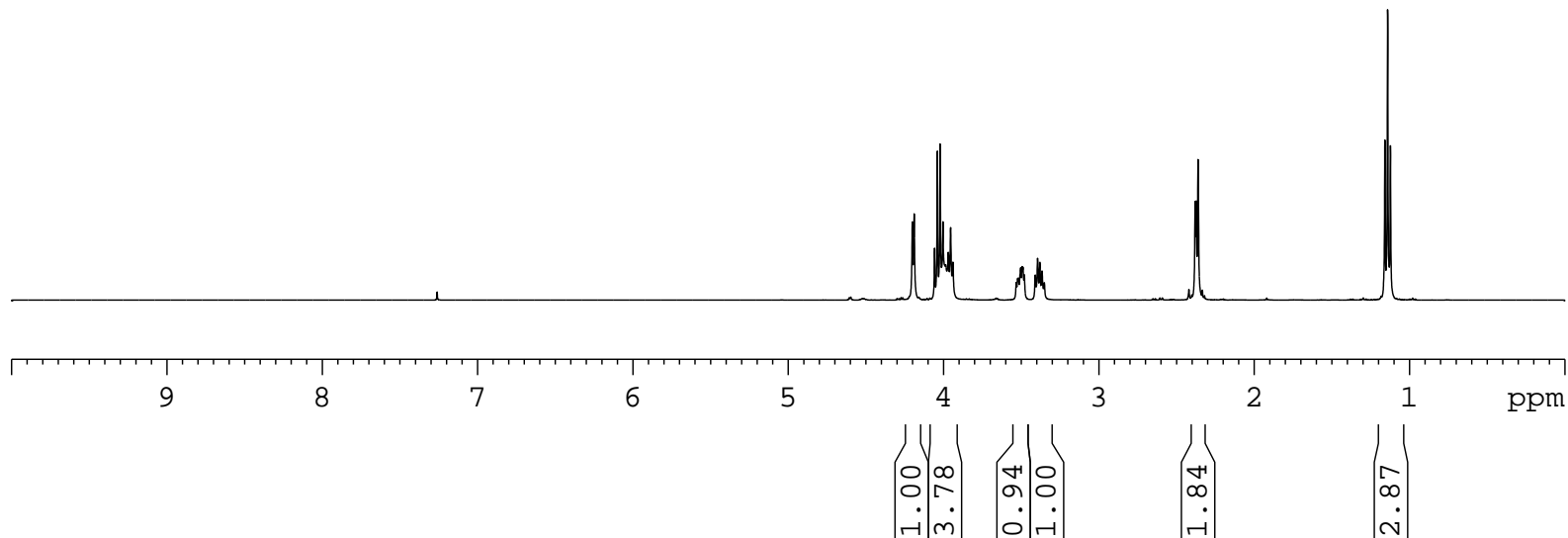


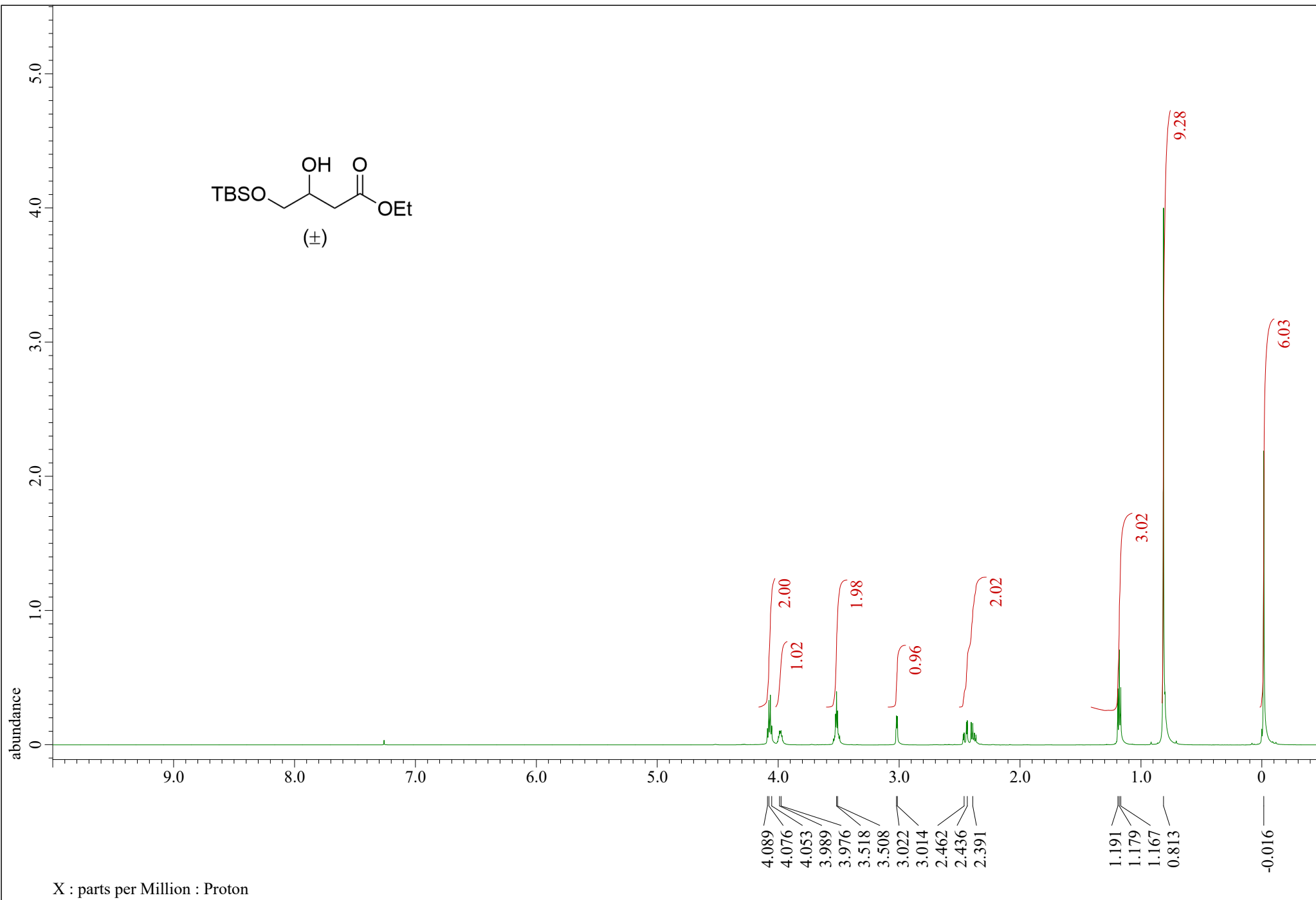
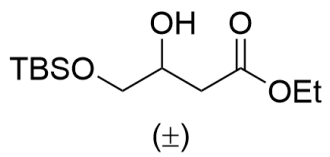
4.2009  
 4.1891  
 4.0576  
 4.0398  
 4.0219  
 4.0040  
 3.9868  
 3.9693  
 3.9545  
 3.9398  
 3.5317  
 3.5229  
 3.5172  
 3.5035  
 3.4943  
 3.4888  
 3.4799  
 3.4096  
 3.3946  
 3.3801  
 3.3660  
 3.3509  
 2.3804  
 2.3736  
 2.3611  
 1.1585  
 1.1407  
 1.1228

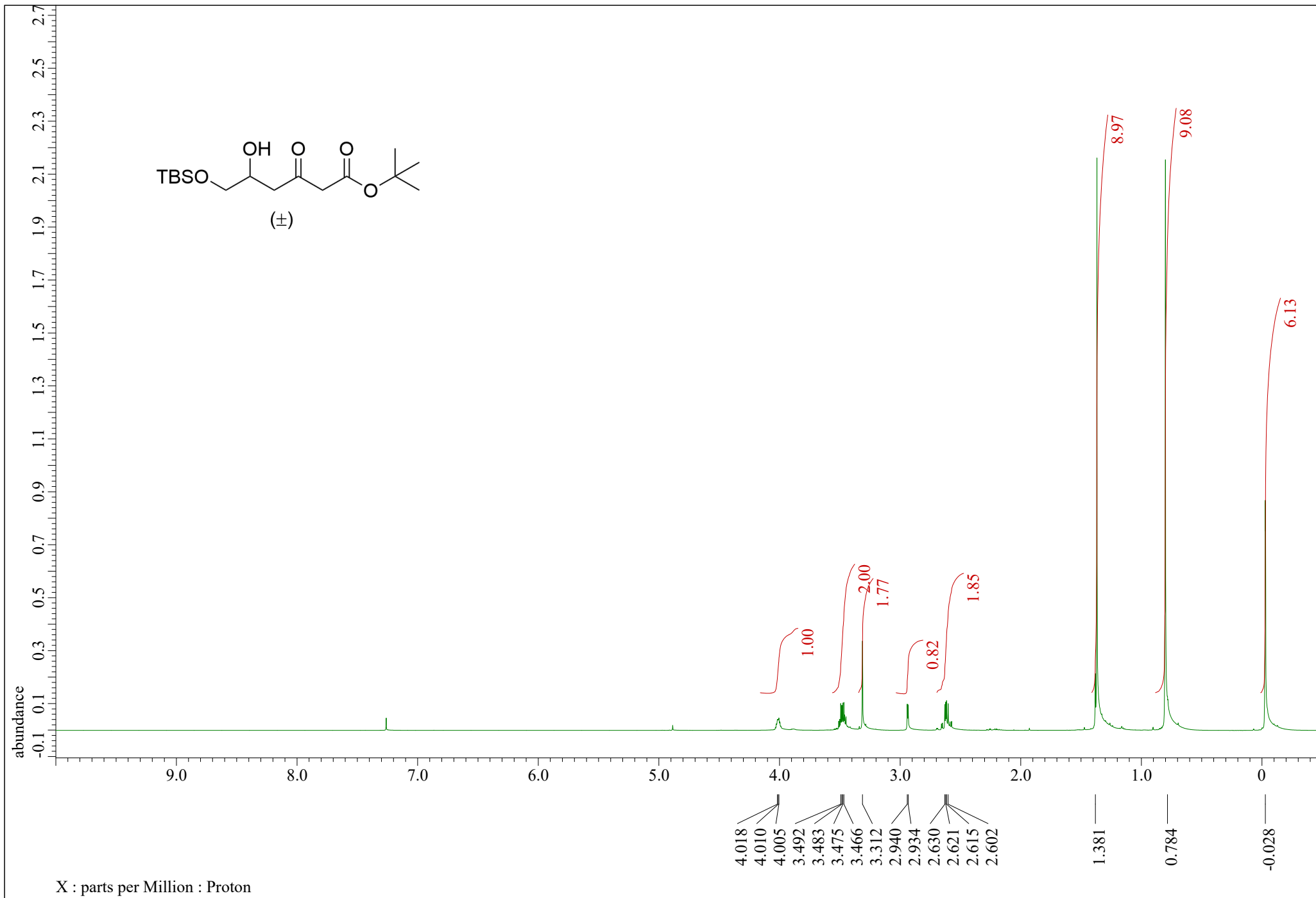


NAME fjj171203  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20171204  
 Time 15.18  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 20.2  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 297.4 K  
 D1 1.00000000 sec  
 TD0 1

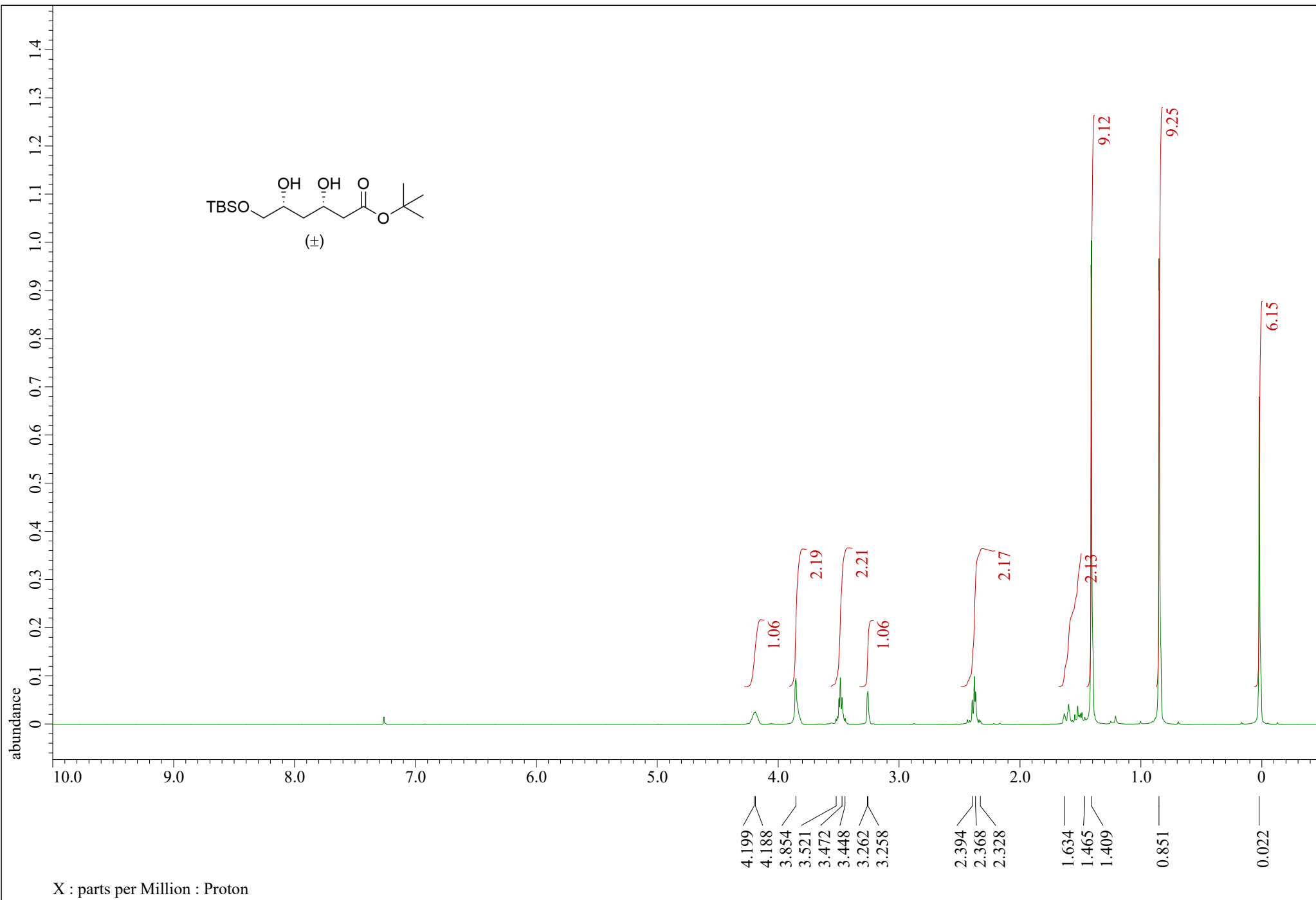
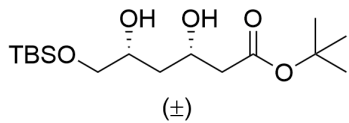
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 15.50 usec  
 PL1 -1.00 dB  
 PL1W 13.18669796 W  
 SF01 400.1724712 MHz  
 SI 32768  
 SF 400.1700020 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

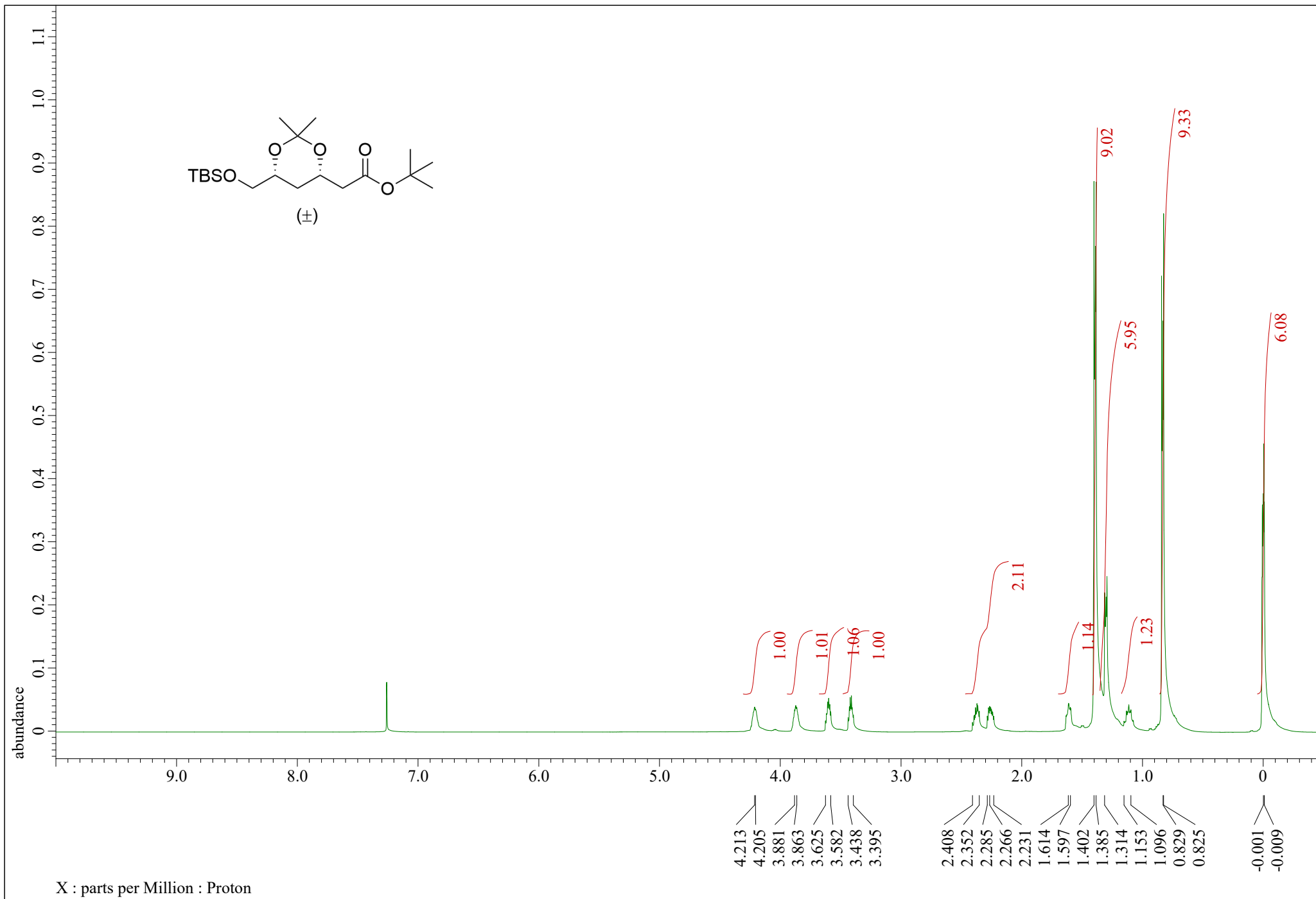


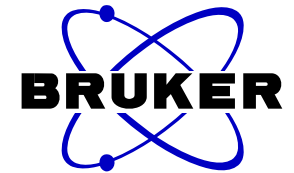
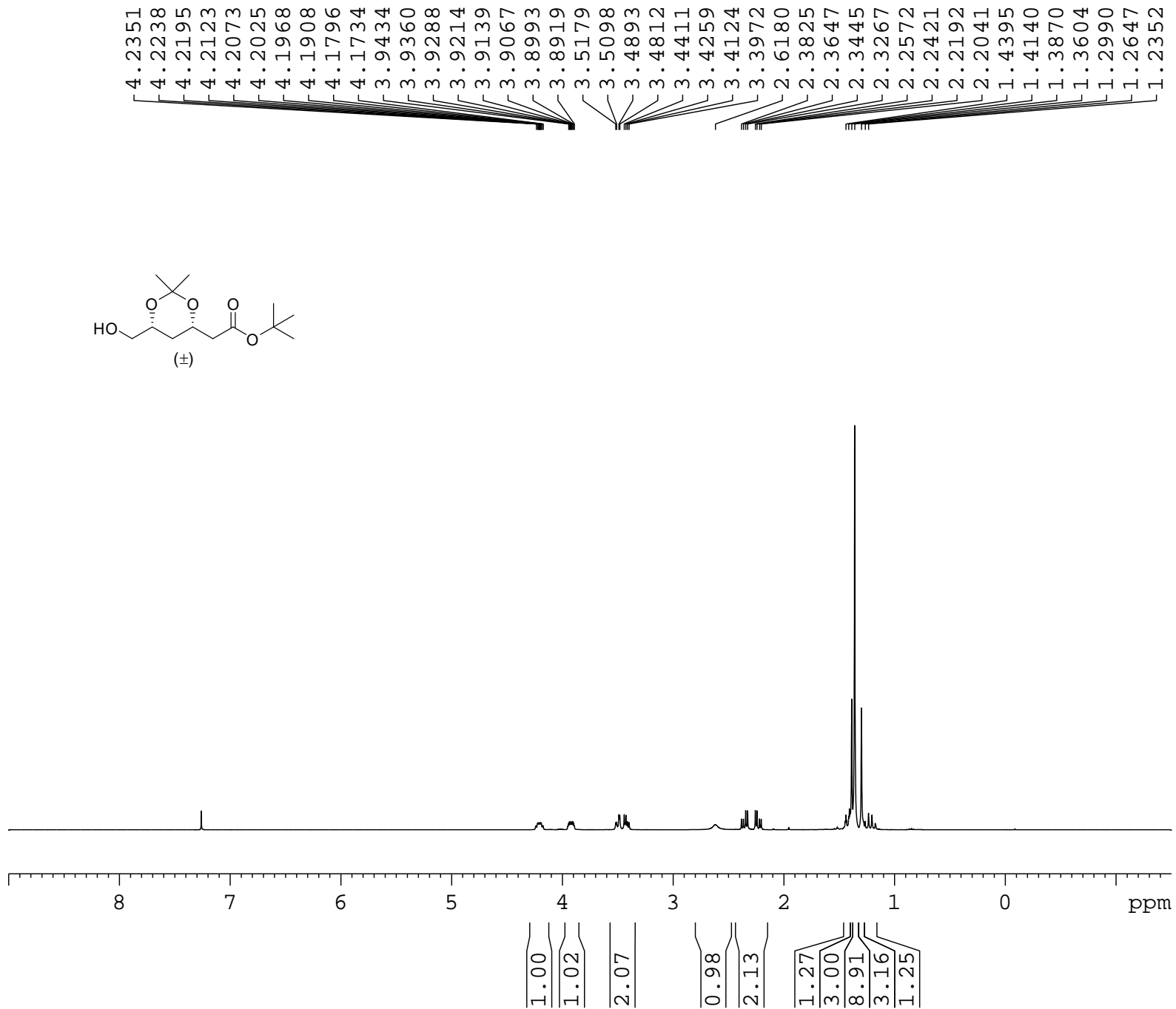
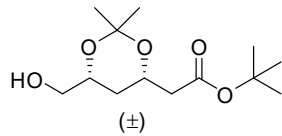








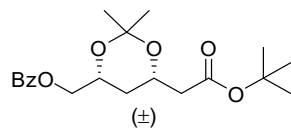




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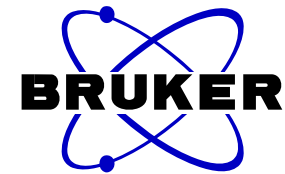
NAME          FJJ171214
EXPNO         1
PROCNO        1
Date_         20171215
Time          14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            22.6
DW            60.800 usec
DE            6.50 usec
TE            294.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           1H
P1             15.50 usec
PL1            -1.00 dB
PL1W          13.18669796 W
SF01          400.1724712 MHz
SI            32768
SF            400.1700023 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.00
  
```



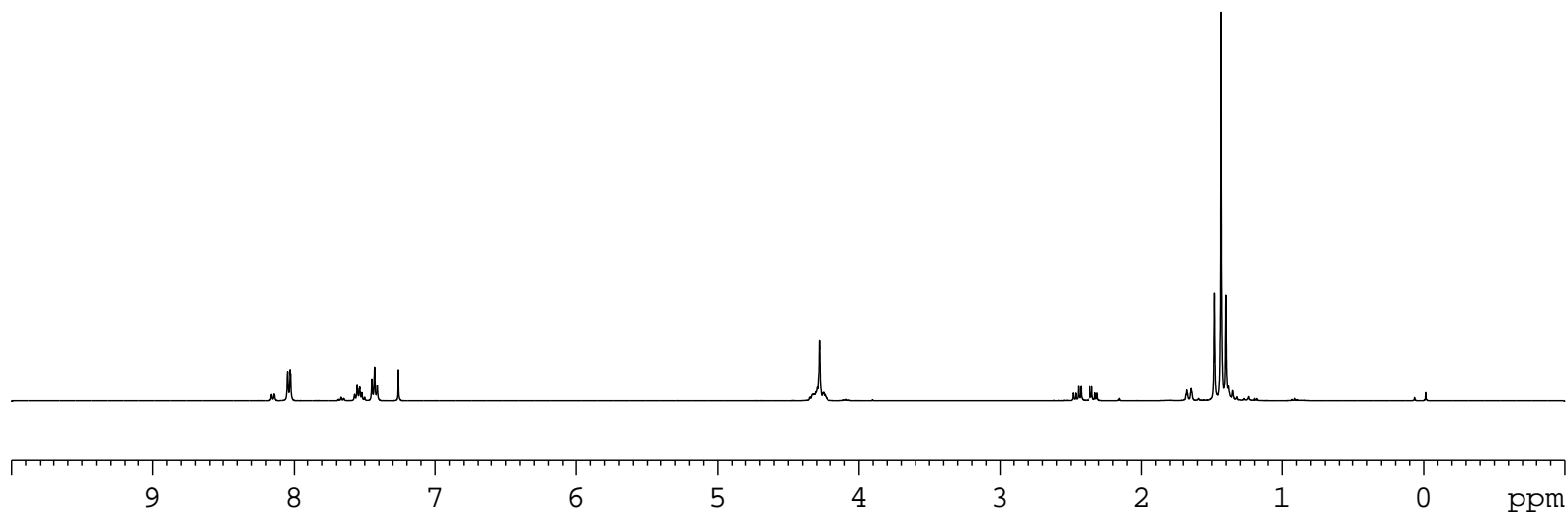
8.0477  
8.0298  
8.0266  
7.5718  
7.5533  
7.5380  
7.5349  
7.5196  
7.4493  
7.4297  
7.4109

4.2800  
2.4838  
2.4660  
2.4460  
2.4282  
2.3634  
2.3485  
2.3256  
2.3106  
1.6809  
1.6753  
1.6696  
1.6437  
1.4810  
1.4346  
1.4011  
1.3835  
1.3676  
1.3537  
1.3234



NAME FJJ171215  
EXPNO 1  
PROCNO 1  
Date\_ 20171215  
Time 19.29  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 50.8  
DW 60.800 usec  
DE 6.50 usec  
TE 292.8 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 15.50 usec  
PL1 -1.00 dB  
PL1W 13.18669796 W  
SF01 400.1724712 MHz  
SI 32768  
SF 400.1700028 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



2.00  
1.34  
2.04

4.02

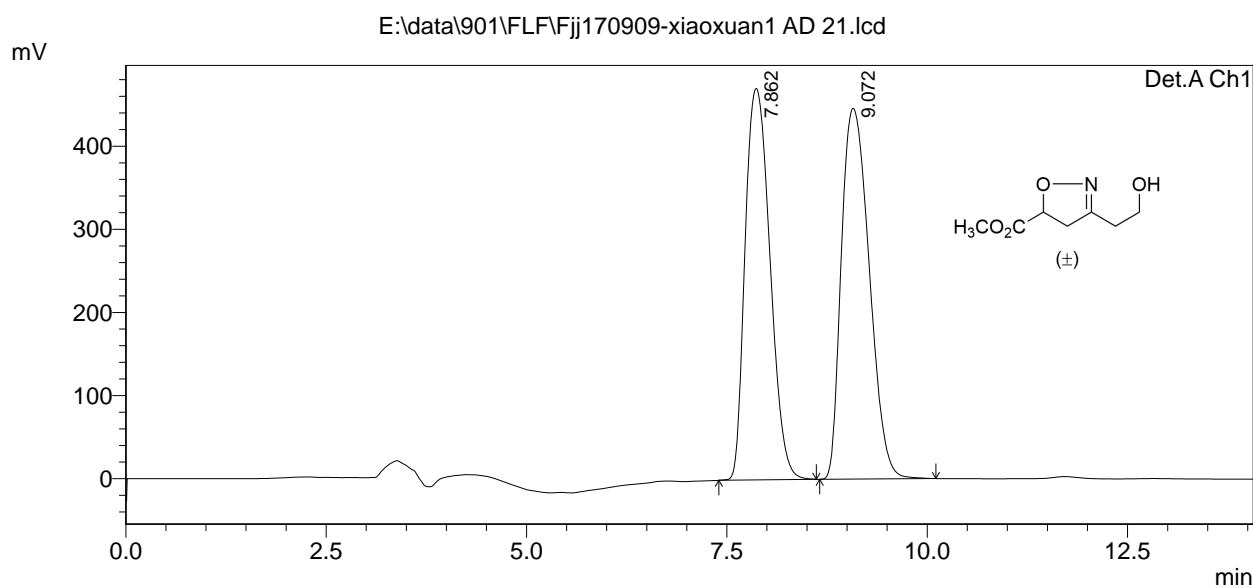
1.99  
1.05  
3.01  
8.57  
2.92  
1.37

# ==== Shimadzu LCsolution Analysis Report ====

E:\data\901\FLF\Fjj170909-xiaoxuan1 AD 21.lcd

Acquired by : Admin  
 Sample Name : Fjj170909-xiaoxuan1  
 Sample ID :  
 Vail # :  
 Injection Volume : 1 uL  
 Data File Name : Fjj170909-xiaoxuan1 AD 21.lcd  
 Method File Name : method1.lcm  
 Batch File Name :  
 Report File Name : Default.lcr  
 Data Acquired : 2017/9/20 10:48:27  
 Data Processed : 2017/9/20 11:02:32

## <Chromatogram>



PeakTable

Detector A Ch1 225nm

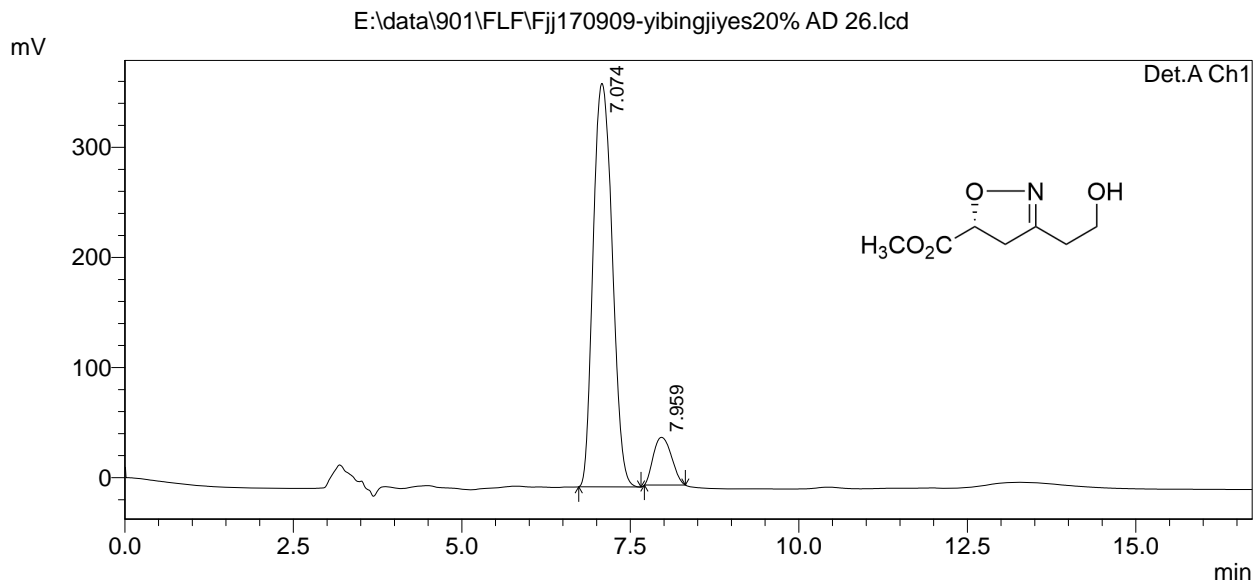
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.862	10179161	470877	48.473	51.351
2	9.072	10820335	446096	51.527	48.649
Total		20999497	916973	100.000	100.000

# ==== Shimadzu LCsolution Analysis Report ====

E:\data\901\FLF\Fjj170909-yibingjiyes20% AD 26.lcd

Acquired by : Admin  
 Sample Name : Fjj170909-yibingjiyes20%  
 Sample ID :  
 Vail # :  
 Injection Volume : 1 uL  
 Data File Name : Fjj170909-yibingjiyes20% AD 26.lcd  
 Method File Name : method1.lcm  
 Batch File Name :  
 Report File Name : Default.lcr  
 Data Acquired : 2017/9/20 16:24:16  
 Data Processed : 2017/9/20 16:41:01

## <Chromatogram>



PeakTable

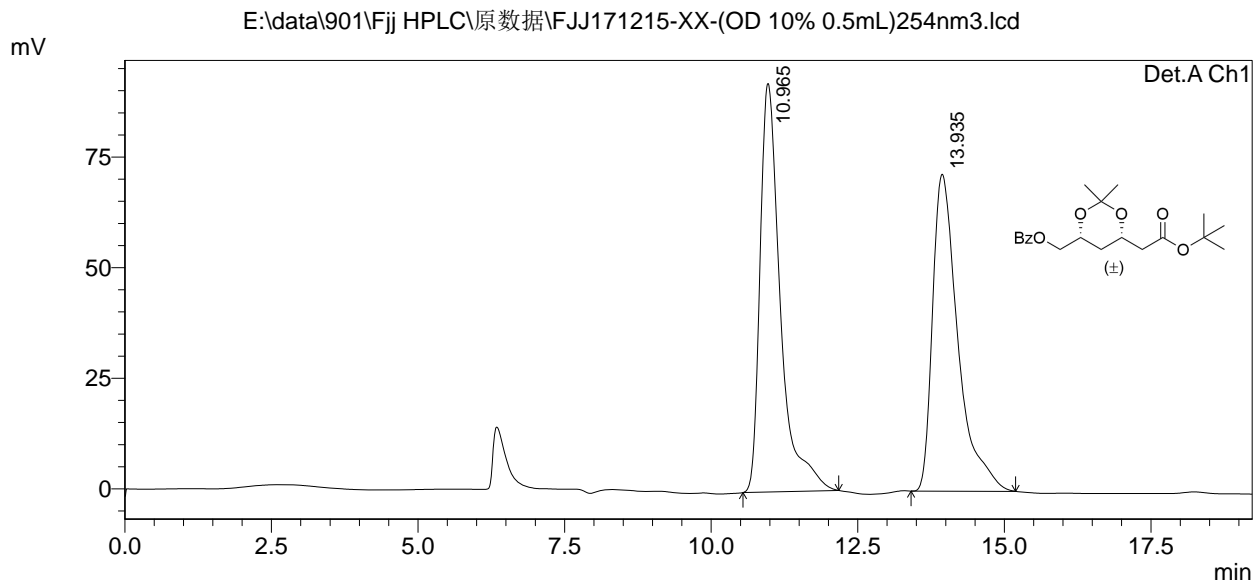
Detector A Ch1 225nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.074	7244825	366410	89.902	89.429
2	7.959	813724	43314	10.098	10.571
Total		8058549	409724	100.000	100.000

# ==== Shimadzu LCsolution Analysis Report ====

E:\data\901\Fjj HPLC\原数据\FJJ171215-XX-(OD 10% 0.5mL)254nm3.lcd  
 Acquired by : Admin  
 Sample Name : FJJ171215-XX-(OD 10% 0.5mL)254nm  
 Sample ID :  
 Vail # :  
 Injection Volume : 1 uL  
 Data File Name : FJJ171215-XX-(OD 10% 0.5mL)254nm3.lcd  
 Method File Name : method1.lcm  
 Batch File Name :  
 Report File Name : Default.lcr  
 Data Acquired : 2017/12/16 18:42:01  
 Data Processed : 2017/12/16 19:50:00

## <Chromatogram>



PeakTable

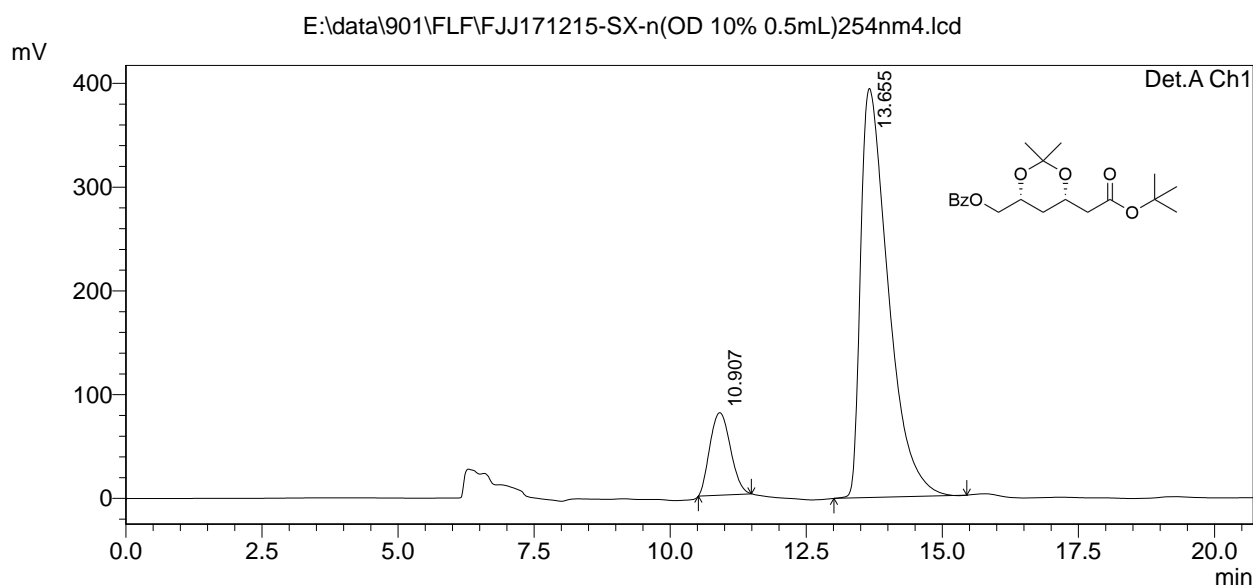
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.965	2232473	92355	50.854	56.314
2	13.935	2157511	71644	49.146	43.686
Total		4389983	163999	100.000	100.000

# ==== Shimadzu LCsolution Analysis Report ====

E:\data\901\FLF\FJJ171215-SX-n(OD 10% 0.5mL)254nm4.lcd  
 Acquired by : Admin  
 Sample Name : FJJ171215-SX-n(OD 10% 0.5mL)254nm  
 Sample ID :  
 Vail # :  
 Injection Volume : 1 uL  
 Data File Name : FJJ171215-SX-n(OD 10% 0.5mL)254nm4.lcd  
 Method File Name : method1.lcm  
 Batch File Name :  
 Report File Name : Default.lcr  
 Data Acquired : 2017/12/16 19:03:28  
 Data Processed : 2017/12/16 19:24:11

## <Chromatogram>



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.907	2097109	79502	13.089	16.779
2	13.655	13924276	394315	86.911	83.221
Total		16021384	473816	100.000	100.000